APR 10 1943

TRANSACTIONS OF THE AMERICAN SOCIETY FOR METALS

MARCH, 1943

olume XXXI

Number 1

The TRANSACTIONS *of the*

AMERICAN SOCIETY FOR METALS

Published quarterly and Copyrighted, 1943, by the AMERICAN SOCIETY FOR METALS
7301 Euclid Avenue, Cleveland, Ohio

SUBSCRIPTIONS: (members) $2.50 per year
(non-members) $5.00 per year, $2.00 per copy
Foreign (non-members) $6.50 per year, $2.50 per copy

Entered as second class matter, November 9, 1931, at the Post Office at Cleveland, Ohio, under the Act of March 3, 1879

RAY T. BAYLESS, *Editor*

Vol. XXXI | March, 1943 | No. 1

The object of the Society shall be to promote the arts and sciences connected with either the manufacture or treatment of metals, or both.

Officers and Trustees

H. J. FRENCH, President
International Nickel Co., New York

M. A. GROSSMANN, Vice-President
Carnegie-Illinois Steel Corp., Chicago

F. B. FOLEY, Treasurer
The Midvale Co., Nicetown, Philadelphia

W. H. EISENMAN, Secretary
7301 Euclid Ave., Cleveland

TRUSTEES

BRADLEY STOUGHTON, Past President
Lehigh University, Bethlehem, Pa.

E. G. HILL
Carnegie-Illinois Steel Corp., Gary, Ind.

V. N. KRIVOBOK
Lockheed Aircraft Corp., Burbank, Cal.

N. F. TISDALE
Molybdenum Corporation of America
Pittsburgh

K. R. VAN HORN
Aluminum Company of America
Cleveland

Publication Committee

M. GENSAMER, Chairman '43
Carnegie Institute of Technology
Pittsburgh

RAY T. BAYLESS, Secretary
7301 Euclid Ave., Cleveland

Members:

J. B. Austin, New York '44
R. M. Brick, New Haven '45
Walter Crafts, Buffalo '44
T. G. Digges, Washington '43
E. H. Dix, Jr., Pittsburgh '43
A. E. Focke, Indianapolis '45
W. R. Frazer, Springfield '45
W. E. Jominy, Detroit '44
E. G. Mahin, Notre Dame '43
Gilbert Soler, Canton-Mass. '44
Clair Upthegrove, Detroit '44
S. P. Watkins, Baltimore '45
T. S. Washburn, Calumet '45
L. L. Wyman, Schenectady '43

TRANSACTIONS
American Society for Metals
VOL. XXXI JANUARY-DECEMBER 1943

DETERMINING THE Ac_3 CRITICAL POINT IN IRON BY A GRAIN ELIMINATION METHOD

By John K. Desmond

Abstract

Large grains which are grown by critically straining iron or alloys of iron and heating it to a temperature above approximately 1200 degrees Fahr. (650 degrees Cent.) persist until a certain higher temperature is reached and maintained. These large grains then disappear. From this fact, the author concludes the Ac_3 critical temperature of iron or its alloys can be determined. Also, in consideration of the theory that these large grains are of free ferrite, it is interesting to record that the noticeable amount of free ferrite is dissolved at one sustained temperature. Perhaps this evidence means that the Ac_3 transformation range is narrower than it is considered generally. The article describes the methods of determining the Ac_3 critical point of an iron-nickel material.

THE known phenomenon of the growth of large ferrite grains in critically strained low carbon iron and low carbon alloys of iron when annealed at a temperature somewhat below their Ac_3 critical point may afford an opportunity to determine by a new method the Ac_3 critical temperatures in these materials. It may also provide evidence for reviewing the Ac_3 reaction.

Fig. 1 demonstrates a block of low carbon nickel-chromium iron, strained by cold hobbing to produce a cavity and annealed at 1250 degrees Fahr. (675 degrees Cent.) which is greatly under its Ac_3 critical point. The iron is shown cut in half vertically through the bottom of the hobbed impression. In certain or critically strained

The author, John K. Desmond, was formerly sales metallurgist of Henry Disston and Sons, Philadelphia, in whose laboratory this work was performed. He is now metallurgist, Ordnance Laboratory, Frankford Arsenal, Philadelphia. Manuscript received January 12, 1942.

areas, large ferrite grains have been developed by the subcritical annealing. They may be observed at some distance from the cavity.

Fig. 2 shows a piece of the same material similarly prepared but annealed at 1600 degrees Fahr. (870 degrees Cent.). The annealing at this temperature, slightly above the Ac_3 point, has removed the

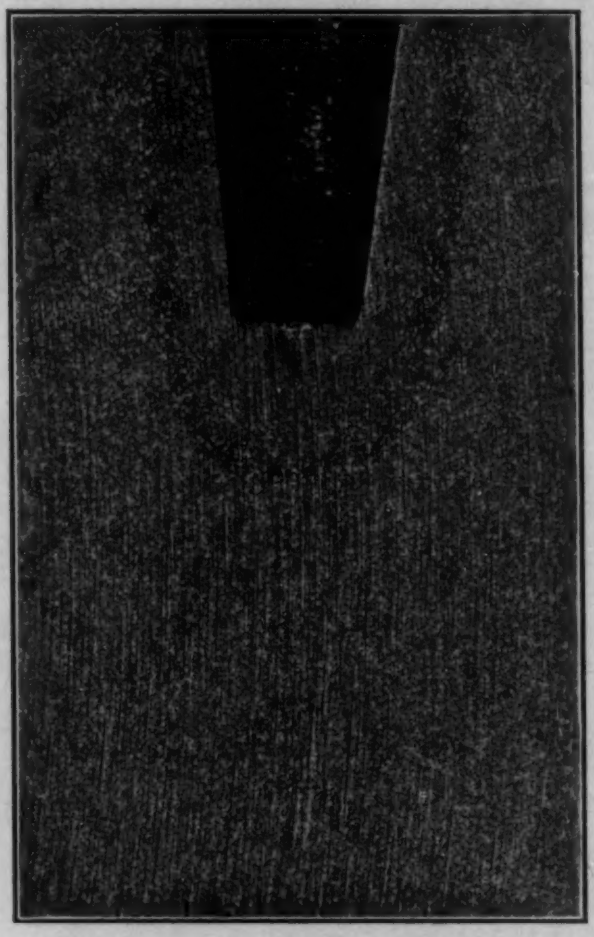


Fig. 1—The Crescent Pattern of Large Ferrite Grains Formed by the Annealing Below the Critical Range.

large ferrite grains previously established at a lower temperature.

The generally accepted understanding of the Ac_3 critical transformation is the temperature range in which ferrite is gradually assimilated into solid solution. In this work we plan to find the lowest temperature capable of eliminating the large grains grown by straining and annealing, and to regard this temperature as the Ac_3 critical transformation for this particular material.

To locate the lowest temperature that might erase these large grains, a number of samples were prepared possessing a distinct design of large grains. The material investigated was a nickel-

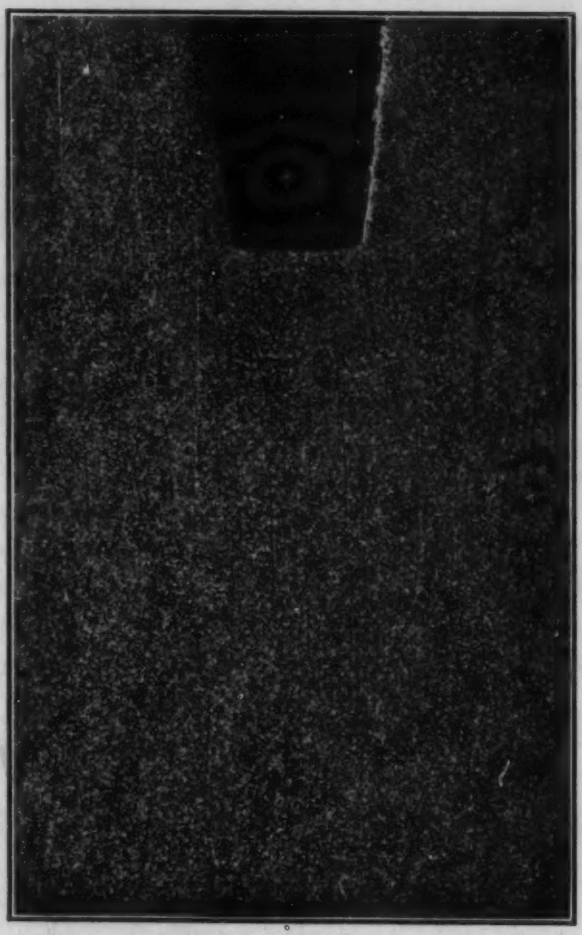


Fig. 2—The Crescent Pattern of Large Ferrite Grains Abolished by the Annealing Above the Critical Range.

chromium iron, used industrially for molds or dies and of the following analysis:

Carbon	Manganese	Silicon	Nickel	Chromium
0.07	0.30	0.14	1.22	0.46

The individual test specimens were made by cold hobbing 1-inch cubes, annealing the blocks at 1250 degrees Fahr. (675 degrees Cent.) for 3 hours and cutting them in half vertically through the bottoms of the cavities. A confirmation of the presence of a ferrite grain arrangement on the sectioned surfaces could be observed by sanding

and swabbing the halves with a 20 per cent solution of ammonium persulphate. The subsequent heating of the test pieces to determine the Ac_3 critical temperature was then carried on in an ordinary oven type electric furnace equipped with a recording potentiometer. An additional instrument with its thermocouple in contact with the pieces, placed in a pipe on the floor of the furnace, registered the temperatures acquired by the specimens. Because of the limits of the furnace control, undesirable fluctuations of 10 degrees Fahr. (5.5 degrees Cent.) were recorded in the test pieces. The maximum temperature of the pieces during these unavoidable oscillations of the furnace was the one adopted for use. With the furnace operating below a supposed Ac_3 critical point for the material, several test specimens were introduced into the pipe in order to determine a reasonable range of temperature in which large grains disappeared. After a practical range of temperature was found for exploration, a new series of test samples was started at the low point of the range. By progressing with 5-degree Fahr. increments, halting at each increase for 1 hour and withdrawing one sample for examination, the lowest temperature was ultimately reached at which the large pre-established ferrite grains were assimilated. Inspection of the test pieces for the presence or absence of the grains was made visually after again sanding and etching.

In the nickel-chromium iron subjected to the treatment described, the large grains remained in specimens annealed at 1560 degrees Fahr. (849 degrees Cent.), fewer were present at 1565 degrees Fahr. (851 degrees Cent.), and all were gone after an annealing at 1570 degrees Fahr. (855 degrees Cent.). The test pieces which retained the large grain formation after a 1560-degree Fahr. (849-degree Cent.) annealing were reheated to 1560 degrees Fahr. (849 degrees Cent.) and held at this temperature for 18 hours. The large grains not only remained after the extended time at temperature but had also increased in size. The test pieces annealed at 1565 degrees Fahr. (851 degrees Cent.) showed a partial assimilation of the ferrite grains at the extremities of the crescent design. These samples were reheated to the same temperature which was held until the ferrite grains disappeared. This required 10 hours to accomplish. We concluded from these results the Ac_3 critical point of this nickel-chromium material to be 1565 degrees Fahr. (851 degrees Cent.).

Several advantages are gained over the usual methods of determining the Ac_3 critical point from the fact that specimens, in this

grain elimination procedure, may be held at stationary temperatures. It is obvious that the specimens may acquire full benefit of each exploratory temperature when they are stopped at that temperature for a certain period of time. The conventional methods for determining critical point reactions require a rapid rate of heating with the temperature of the specimen continuously rising. As is well known, a continuously increasing rate of heating may be a varying

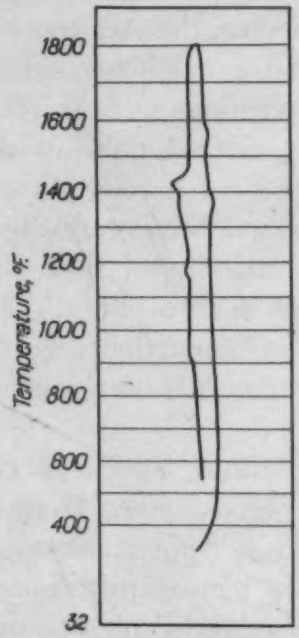


Fig. 3 — LeChatlier-Saladin Heating Curve (Right) and Cooling Curve (Left) of the Iron-Nickel Material Used in This Work. (Courtesy Francis B. Foley, Midvale Co.)

influence, not only in altering the position at which the Ac_3 transformation is due, but also in producing a lag in the Ac_3 maximum manifestation. In this suggested system of eliminating the grains, the rate of heating which might ordinarily produce these effects stops naturally when the specimens arrive at the fixed furnace temperature. At this stabilized point, the metal should adjust itself to the temperature. If due, the Ac_3 reaction will occur.

In this experimental work we have been able to preserve intact a noticeable amount of free ferrite until a certain temperature is reached during heating. We have observed a growth of the ferrite grains in the specimens heated below this temperature. In addition, we find that within a narrow temperature range of 5 degrees Fahr.

the subcritically grown massive ferrite grains remain either indissoluble or can be dissolved. From this evidence, we believe it may be assumed that other unobserved free ferrite enters solution at the same time as the large grains. If our evidence is correct and our assumption warranted, the Ac_3 critical point is the beginning and the end of a reaction occurring at one particular temperature.

Fig. 3 represents a LeChatelier-Saladin heating curve (right) and cooling curve (left) of the iron-nickel material used in this work. From the heating curve, the Ac_3 transformation range would be regarded as starting in the neighborhood of 1520 degrees Fahr. (825 degrees Cent.) and terminating near 1600 degrees Fahr. (870 degrees Cent.). The Ac_3 critical point would be placed at 1575 degrees Fahr. (855 degrees Cent.). At this temperature the bulk of the ferrite is considered to have been gradually assimilated. It seems extraordinary that a large amount of heat, depicted by the jog on the heating curve at 1560 degrees Fahr. (850 degrees Cent.) is needed near the terminus of a reaction regarded usually as gradual and continuous. This paradox has been noticed and called to attention by others.

In the opinion of the author, the curious appearance of the jog on the heating curve might be explained from our observations made in eliminating ferrite grains. Appreciable amounts of ferrite were dissolved within a narrow temperature range, the Ac_3 point, at which we would expect a considerable absorption of heat. Other aspects of the heating curve we might also plausibly interpret from our work with critically strained iron. In our specimens there would be a heat absorption during the temperature range in which the free ferrite was growing. In the specimen represented by the thermocurve, the heat absorption manifested from 1515 to 1560 degrees Fahr. (825 to 850 degrees Cent.) might possibly be considered as the heat required to relieve the metal of its ordinary strains. It is, of course, impossible to have any specimen entirely free of strains.

We are inclined to regard the terminus of the Ac_3 reaction at the temperature where the large grains disappeared. The sloping back from 1560 degrees Fahr. (850 degrees Cent.) to 1600 degrees Fahr. (870 degrees Cent.), which the thermocurve shows, can be explained, we think, by the fact that the specimen could not be held stationary at 1560 degrees Fahr. (850 degrees Cent.) for a length of time sufficient for the reaction to be consummated.

In conclusion, we believe this grain elimination method is a new way of determining the Ac_3 transformation in low carbon iron and alloys of iron; that the procedure is simple and capable of duplication of results, and that from our work we have found the Ac_3 transformation to develop within a very narrow temperature range.

CARBURIZING CHARACTERISTICS OF 0.20 PER CENT CARBON ALLOY AND PLAIN CARBON STEELS

By G. K. Manning

Abstract

Eight common carburizing steels were tested in five different commercial solid carburizers.

Chromium steels produced cases with extremely high surface carbon content when carburized in four of the carburizers. Only one of the carburizers tested resulted in low surface carbon concentration for chromium steels. Nickel opposed chromium in its effect on surface carbon concentration.

Contrary to a common belief, lower carburizing temperatures did not result in cases of greater surface carbon content.

A low carbon skin (0.0003 to 0.001 inch deep) was observed to be present after carburizing for 10 hours or longer at 1700 degrees Fahr. (925 degrees Cent.) but was not found at lower carburizing temperatures.

Introduction

PRESENT conditions made it necessary for consumers of carburizing steels to change to new steels containing smaller amounts of alloying elements or a new combination of alloying elements. Many consumers had had experience with only one or two carburizing steels and the changes made necessary by the country's efforts to conserve certain of the alloying elements naturally gave rise to questions about the comparative carburizing characteristics of different grades. In particular, the national emergency pushed the chromium-molybdenum type of carburizing steel into prominence. Piecemeal evidence indicated that chromium-molybdenum steels under some conditions, at least, gave unusually high carbon concentration at the surface. For some applications high carbon cases might be advantageous; but in many instances (e.g. clash gears) the thought of surface carbon concentration of 2 per cent and higher was sufficient to cause concern among consumers. One

A paper presented before the Twenty-fourth Annual Convention of the Society, held in Cleveland, October 12 to 16, 1942. The author, G. K. Manning, is associated with the metallurgical department, Republic Steel Corp., Chicago. Manuscript received June 10, 1942.

large consumer felt that high surface carbon content might be avoided by limiting the chromium content of the S.A.E. 4120 type steels to 0.65 per cent, the manganese being raised to compensate for lowering the chromium. Makers of carburizing compounds suggested that the surface carbon concentration might be controlled by proper choice of carburizing compound.

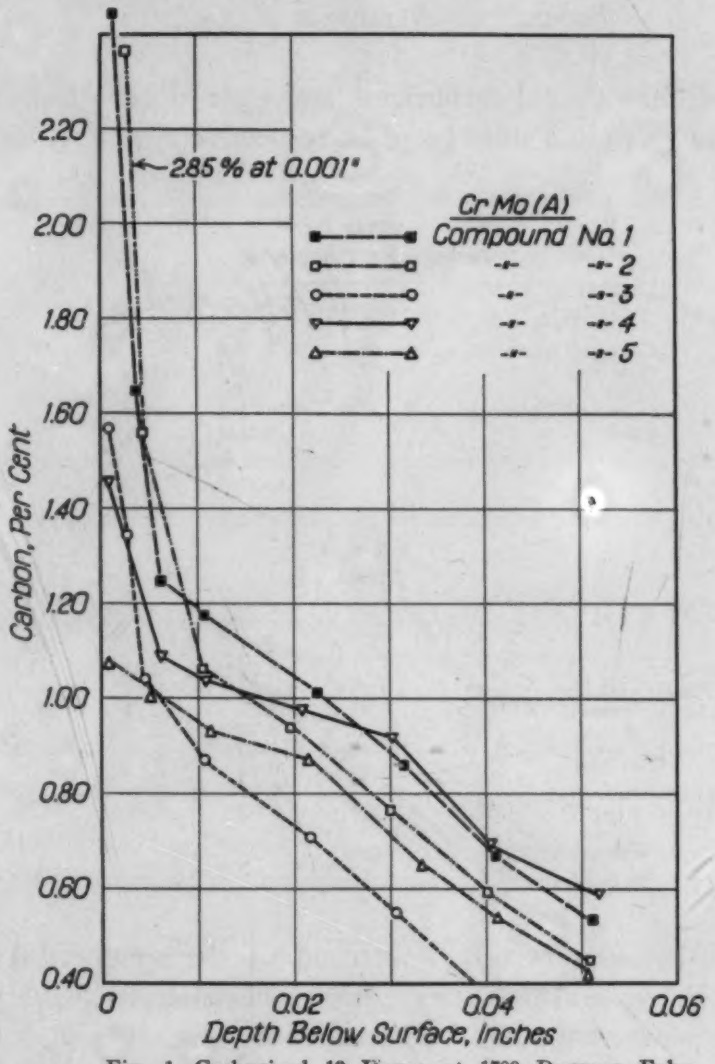


Fig. 1—Carburized 10 Hours at 1700 Degrees Fahr. Air-Cooled in Box.

The present work was started in an effort to obtain more detailed information about such problems as these. As the tests progressed and the results were obtained new questions presented themselves until, eventually, information was obtained about all the principal variables encountered in commercial pack carburizing.

One advantage of dealing with all the variables in the same experiment was that the same materials, equipment and technique could be used throughout, and any errors encountered became at least consistent errors and, as such, were not likely to mask the effect of the variables being considered.

Effect of Alloying Elements

Five different solid carburizers and eight different steels, whose analyses are given in Tables I and II, respectively, were chosen for the

Table I
Carburizing Compounds

Number	Base	Composition in Per Cent Na	Ca	Ba	Fe
1	Coke and Charcoal	1.1	2.6	7.0	...
2	Charcoal	2.7	0.3	...	...
3	Coke	...	2.7	0.7	7.0
4	Coke and Charcoal	2.3	0.5	0.1	...
5	Coke	...	...	15.0	...

Table II
Steels

Steel	Composition in Per Cent C	Si	Mn	Ni	Cr	Mo
Plain Carbon	0.20	0.17	0.86	0.08	0.07	....
1.75% Nickel-Molybdenum	0.20	0.19	0.42	1.65	0.04	0.20
3.5% Nickel-Molybdenum	0.20	0.23	0.55	3.58	0.04	0.25
3.5% Nickel	0.21	0.23	0.53	3.54	0.03	....
Chromium-Nickel	0.19	0.21	0.63	1.32	0.53	....
Chromium	0.20	0.18	0.44	0.13	0.70	....
Chromium-Molybdenum (A)	0.17	0.24	0.68	0.08	1.06	0.26
Chromium-Molybdenum (B)	0.26	0.19	0.93	0.08	0.56	0.16

All steels were fine-grained aluminum-killed.

tests. The carburizers tested were all of the commercial type intended to be replenished after each carburizing cycle with a percentage of new compound. The ratio of new and old compounds mixed in commercial practice varies widely. To test all the various ratios of old and new compounds which might be used would lead to an almost endless amount of testing. Consequently the testing was limited to the use of 100 per cent new compound. Specimens were turned and polished before carburizing, then carburized, recentered in the lathe with an indicator, and turnings collected for chemical analysis at appropriate depths below the surface. Details

of the operations together with a complete outline of the exact carburizing cycles used may be found in the appendix. Figs. 1 to 8 inclusive are graphs of results obtained by carburizing all eight steels in each of the five compounds using 10 hours at 1700 degrees Fahr. (925 degrees Cent.).

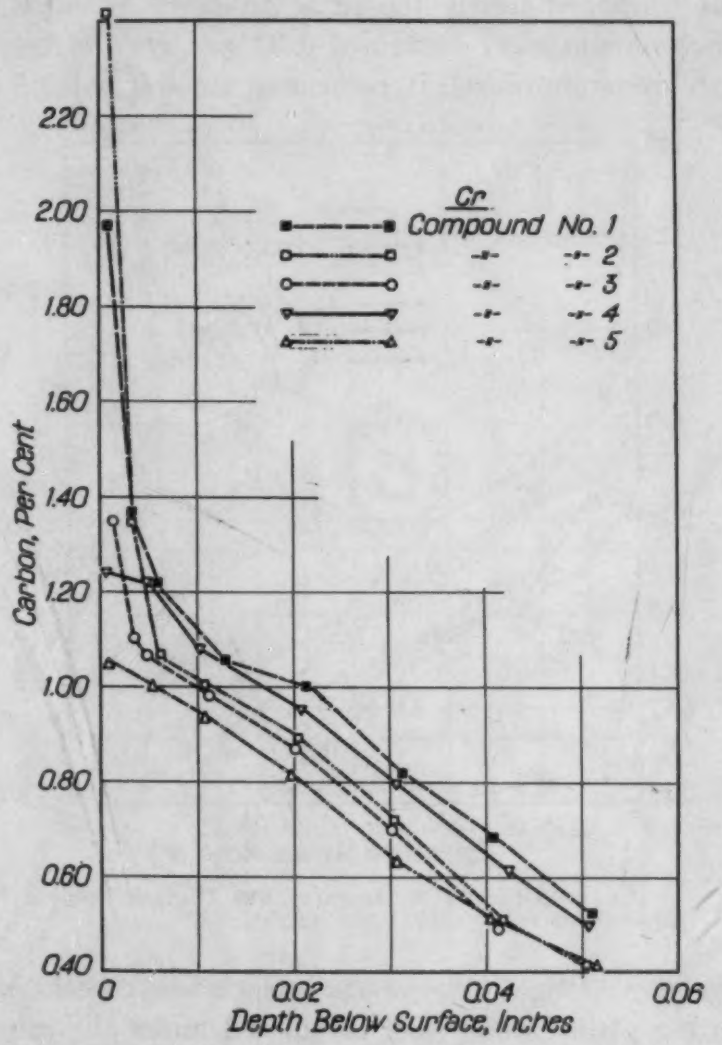


Fig. 2—Carburized 10 Hours at 1700 Degrees Fahr. Air-Cooled in Box.

A. Chromium: For convenience Figs. 1 to 4 are arranged in order of decreasing chromium content. It can be seen that chromium, a strong carbide-forming element, may lead to extreme carbon concentrations at the surface. All the chromium alloy steels carburized 10 hours at 1700 degrees Fahr. (925 degrees Cent.) in compounds No. 1 and No. 2 had excess carbides present near the surface which

tended to be globular in form. In the absence of nickel the higher the chromium content, the higher was the surface carbon content of the case when carburized in either compound No. 1 or No. 2.

B. Nickel: The presence of nickel tended to modify the effect of chromium. This effect can be noted by comparing Figs. 3 and 4. Both steels contained nearly the same amounts of chromium, but the nickel-chromium steel contained 1.32 per cent nickel. In the absence of chromium, nickel, if present in amounts of 3.5 per cent,

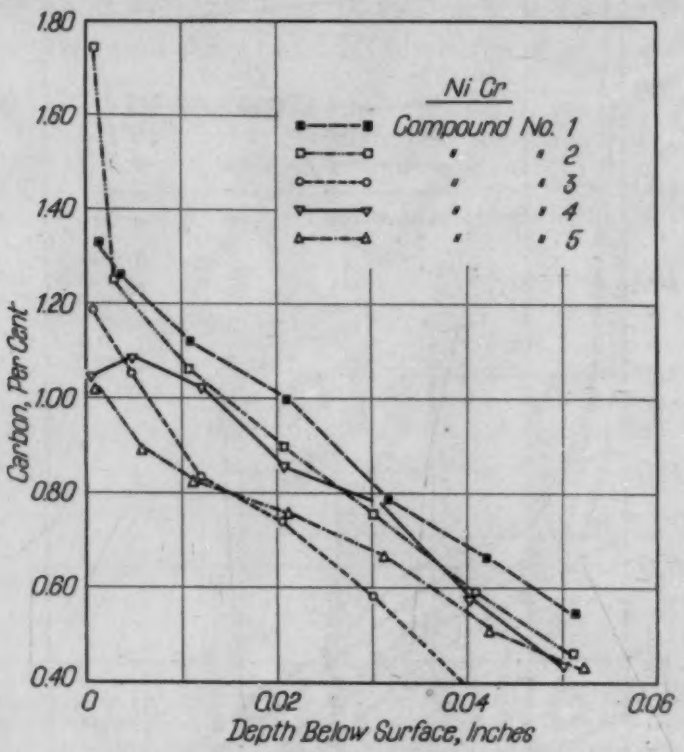


Fig. 3—Carburized 10 Hours at 1700 Degrees Fahr. Air-Cooled in Box.

appeared to give a slightly lower surface carbon content than that obtained in the plain carbon steel carburized under the same conditions. Compare Fig. 5 with Figs. 6 and 8.

C. Molybdenum: A comparison of the case produced in the 3.5 per cent nickel steel (Fig. 6) with that produced in the 3.5 per cent nickel-molybdenum steel (Fig. 8) indicated that the presence of 0.25 per cent molybdenum tended to give a slightly lower surface carbon content. The effect was, however, quite small and further confirmation would be desirable.

D. Other elements: The variation in amounts of the other ele-

ments (manganese, silicon) contained in the steels was not sufficiently great to afford a conclusion as to the effect of these elements.

E. Case depth, which is substantially the same for all steels, appeared to be largely independent of the alloying elements present.

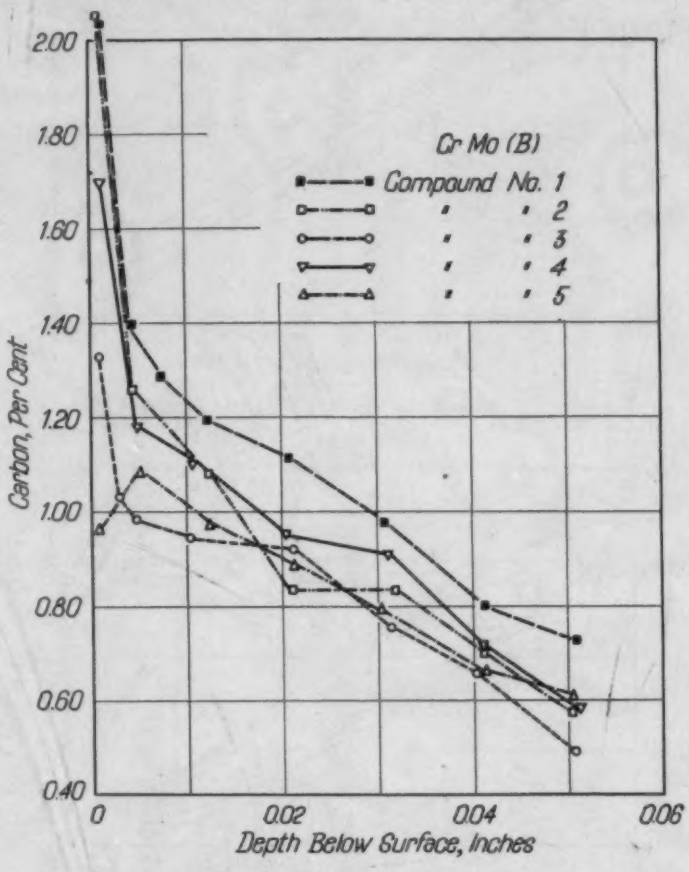


Fig. 4—Carburized 10 Hours at 1700 Degrees Fahr. Air-Cooled in Box.

Effect of Compound

Figs. 1 to 8 inclusive also serve to illustrate the variations in surface carbon concentrations and case depth which were obtained from different carburizers.

A. Surface Carbon: No. 1 compound and No. 2 compound resulted in extremely high carbon surfaces for the chromium steels. (Figs. 1 to 4). Surfaces of these steels ranged from 1.37 to 2.85 per cent carbon with these compounds. No. 3 compound and No. 4 compound gave surface carbon contents intermediate between the high results obtained with No. 1 compound or No. 2 compound and the low results obtained with No. 5 compound. The only

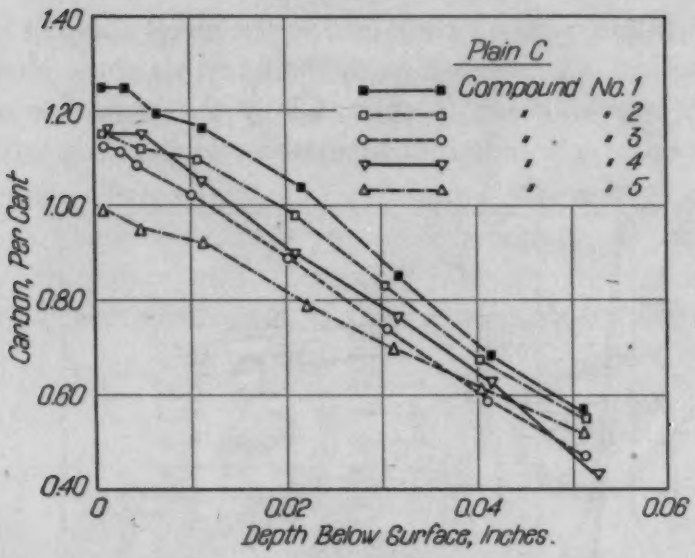


Fig. 5—Carburized 10 Hours at 1700 Degrees Fahr. Air-Cooled in Box.

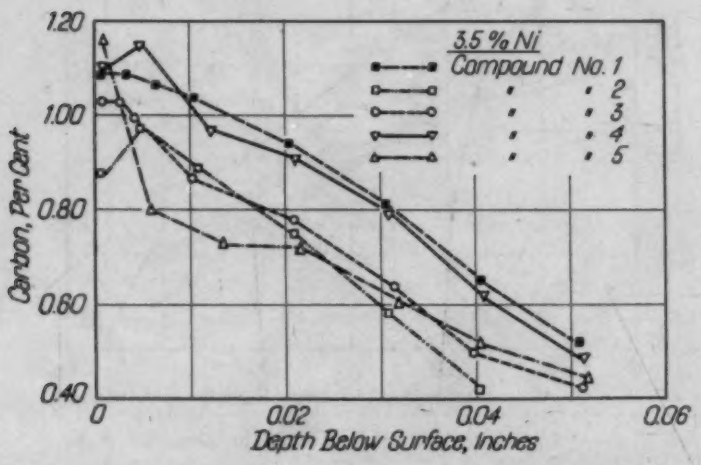


Fig. 6—Carburized 10 Hours at 1700 Degrees Fahr. Air-Cooled in Box.

compound which produced low carbon cases for all steels was the No. 5 compound. The extreme surface of the case produced by carburizing the chromium-molybdenum (A) steel for 10 hours at 1700 degrees Fahr. (925 degrees Cent.) in No. 5 compound and cooling in the box in air is shown in Fig. 9. The carbide network present is such as to indicate approximate saturation of the austenite prior to cooling. The surface carbon content of the remaining steels (plain carbon, 1.75 per cent nickel-molybdenum, 3.5 per cent nickel, and 3.5 per cent nickel-molybdenum) remained below 1.25 per cent regardless of the carburizer used. One conspicuous feature of many of the curves was that the carbon content reached a maximum

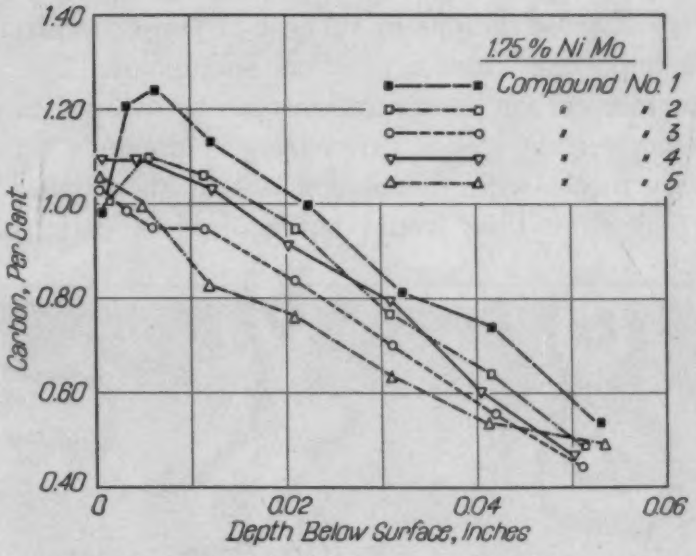


Fig. 7—Carburized 10 Hours at 1700 Degrees Fahr. Air-Cooled in Box.

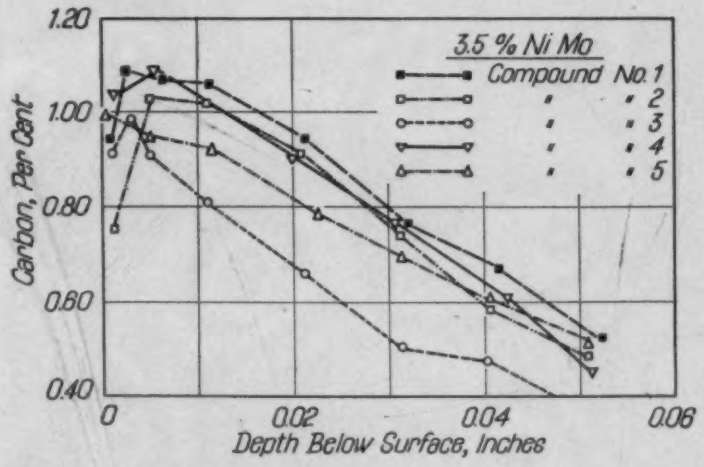


Fig. 8—Carburized 10 Hours at 1700 Degrees Fahr. Air-Cooled in Box.

at a certain depth below the surface. For the 3.5 per cent nickel-molybdenum steel, this effect was apparent by chemical analysis in four of the six curves. See Fig. 8. Fig. 9 also gave evidence of the same condition. The carbide network did not extend to the surface.

B. Case Depth: It will be noted that variations in case depth brought about by the use of different compounds were small. In general, it can be said that No. 1 compound produced a slightly deeper case than the other compounds. No. 2, No. 4 and No. 5 compounds all gave approximately the same case depth. Results obtained with No. 3 compound were not consistent. The fact that only relatively small variations in case depth were encountered,

although very large variations in surface carbon concentrations were obtained, is important from a practical standpoint. The suggestion that high surface carbon concentrations can be avoided in chromium steels by using certain special carburizing compounds (such as No. 5) sometimes meets with the objection that these compounds are slower so that more time would be required in carburizing other

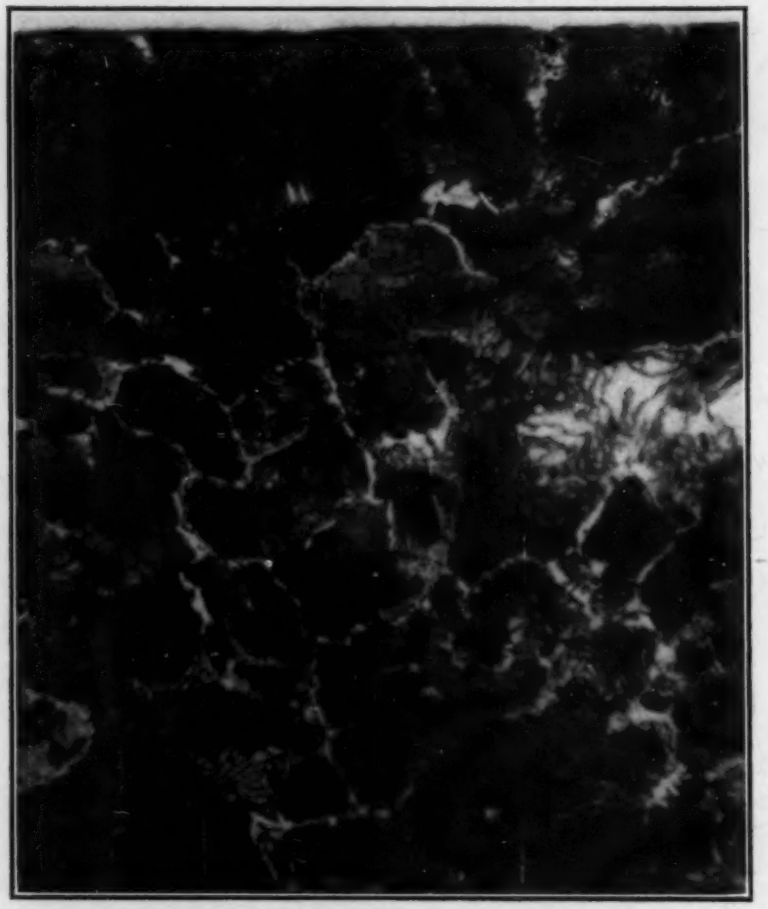


Fig. 9—Chromium-Molybdenum Steel (A) Carburized 10 Hours in Compound No. 5 at 1700 Degrees Fahr. Cooled in Carburizing Box in Air. Top of Photomicrograph was Surface at Time of Carburizing. × 1000.

grades of steel. The use of two different compounds in the same shop is naturally objectionable. The results obtained in this investigation indicate that no substantial increases in carburizing time would be required if compound No. 5 were used for all of the grades of steel. All tests were made in new compound, so that no evidence was obtained regarding the life, shrinkage and relative economy of the various compounds used.

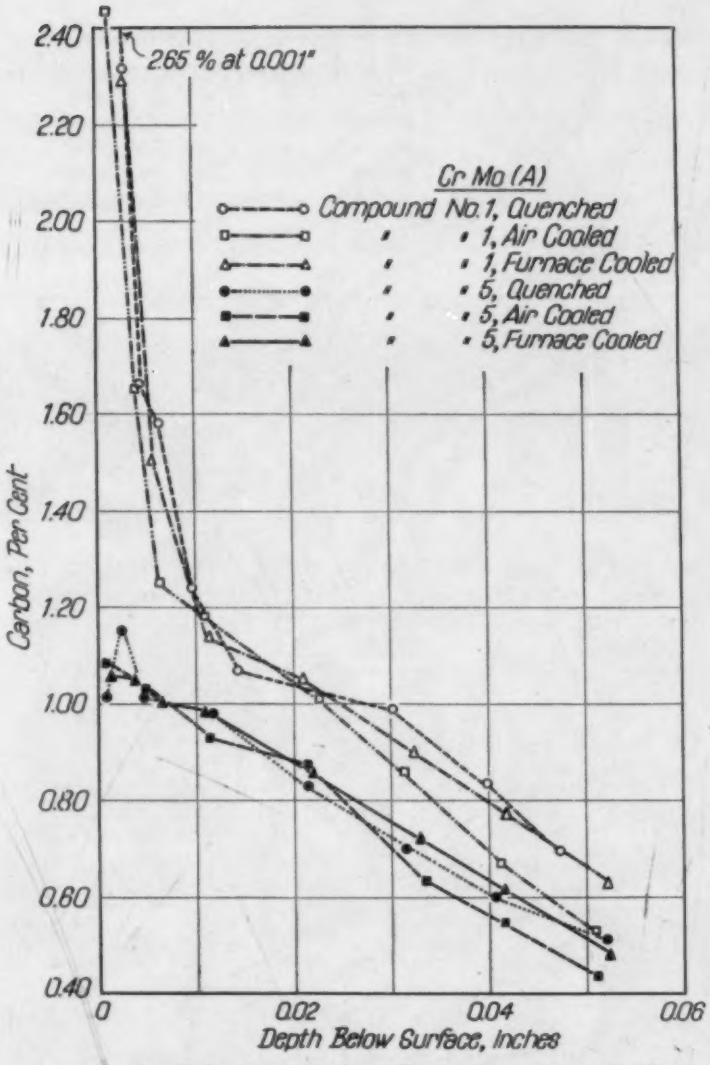


Fig. 10—Effects of Cooling Rate, Carburized 10 Hours at 1700 Degrees Fahr.

Questions, which arose from finding that by both the microscope and chemical analyses the maximum carbon concentration was reached some distance below the surface, led to additional tests. Was this low carbon skin due to simple decarburization during cooling from the carburizing temperature? If so, then direct quenching should eliminate it and furnace cooling should make it much deeper. Was it due to exhaustion of the carburizing compound? In this event longer carburizing times should cause it to increase in depth.

Effect of Cooling Rate

Three steels, chromium-molybdenum (A), nickel-chromium and the plain carbon were carburized in the two compounds which

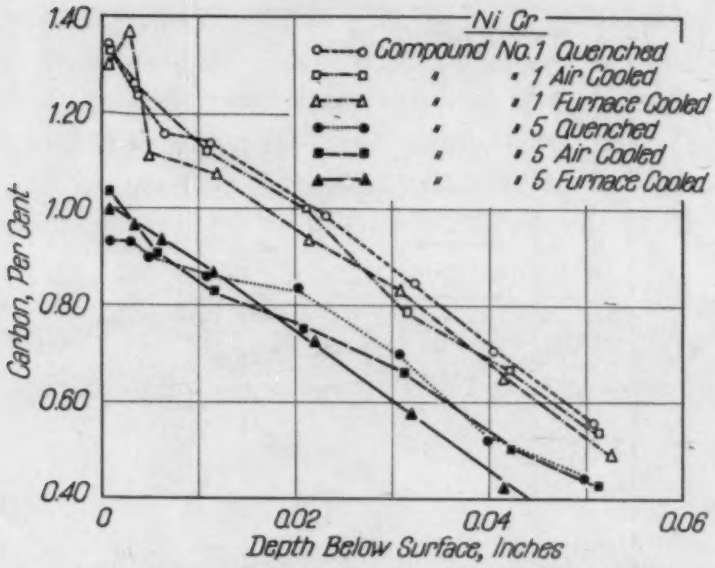


Fig. 11—Effects of Cooling Rate, Carburized 10 Hours at 1700 Degrees Fahr.

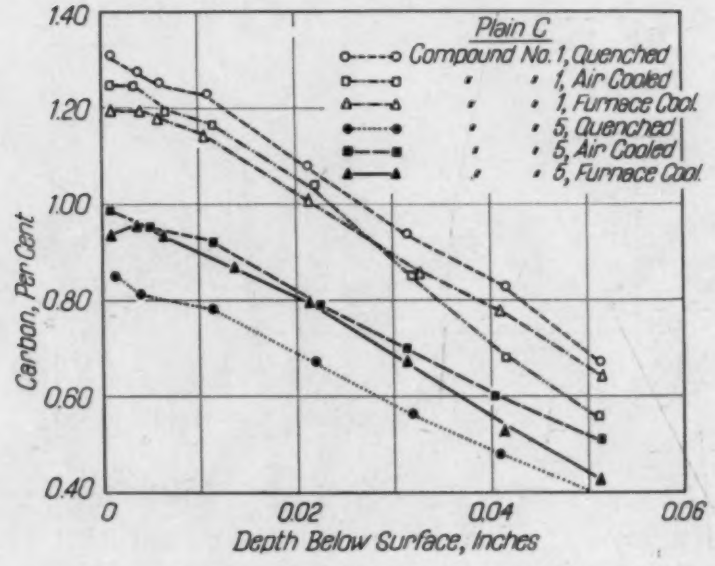


Fig. 12—Effects of Cooling Rate, Carburized 10 Hours at 1700 Degrees Fahr.

had produced the greatest difference in surface carbon concentration, No. 1 and No. 5. Separate sets were quenched direct and furnace-cooled from the carburizing temperature. Results are shown in Figs. 10, 11 and 12.

A comparison of the graphs indicated a maximum spread of 0.14 per cent carbon at any given depth below the surface for a particular steel, with a given time, temperature and compound. Furthermore, it was noted from inspection of Figs. 10 to 12 that the

difference in carbon analyses between similar samples cooled differently was not consistent. It was, therefore, concluded that the effect of cooling velocity on carbon content of the case was so small that it was obscured by other effects such as reproducibility of the method and errors in chemical analysis. Microscopic examination had to be employed to determine the effect of cooling rate on the

Fig. 13—Chromium-Molybdenum Steel (A) Carburized 10 Hours in Compound No. 1 at 1700 Degrees Fahr. Quenched from the Carburizing Temperature and Drawn 3 Minutes in Lead at 1225 Degrees Fahr. Top of Photomicrograph was Surface at Time of Carburizing. × 1000.

low carbon skin. Examination indicated that this skin was present on all the specimens which contained excess carbides regardless of whether the specimens were quenched or furnace-cooled. Steels carburized in compound No. 5 had no excess carbides present after quenching and therefore evidence concerning the low carbon skin was lacking for these specimens. From the condition found at

the surface of the chromium-molybdenum (A) steel direct quenched, it appeared that the low carbon skin was formed during the carburizing cycle, not during cooling from that temperature. Fig. 13.

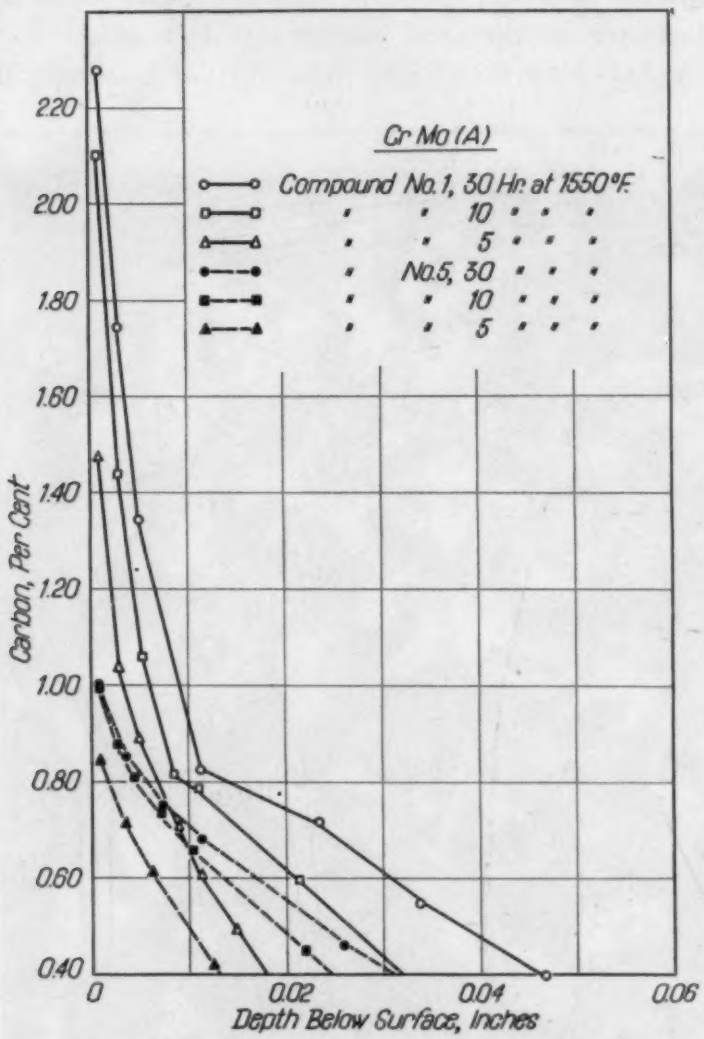


Fig. 14—Effects of Time at 1550 Degrees Fahr. Quenched from Carburizing Temperature.

Figs. 10 to 12 served as a means of judging the errors which might be expected from sampling, chemical analysis, variations in analysis of the carburizing compounds, and possibly even differences in the degree of homogeneity in the specimens themselves. While it was evident that the curves did not have the reproducibility which might be desired for very precise work, it was also evident that the degree of reproducibility which could be expected was sufficiently good to justify the conclusions to be drawn.

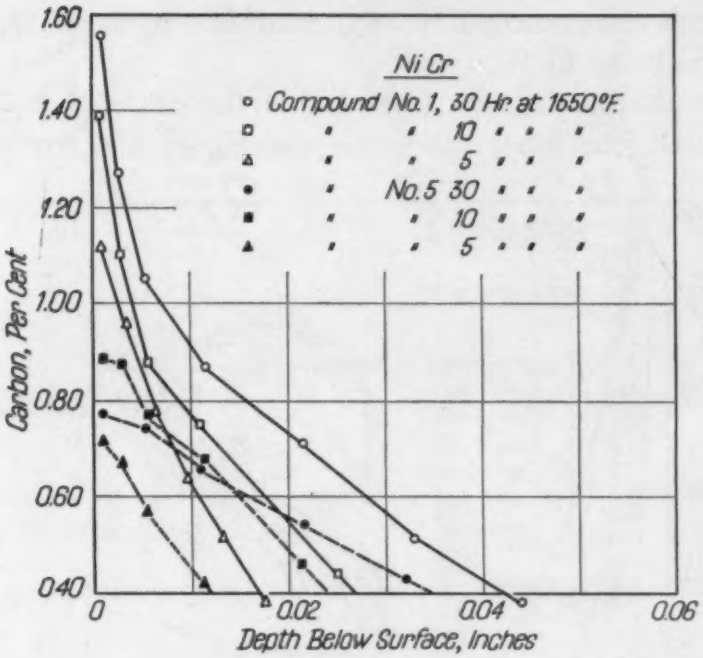


Fig. 15—Effects of Time at 1550 Degrees Fahr. Quenched from Carburizing Temperature.

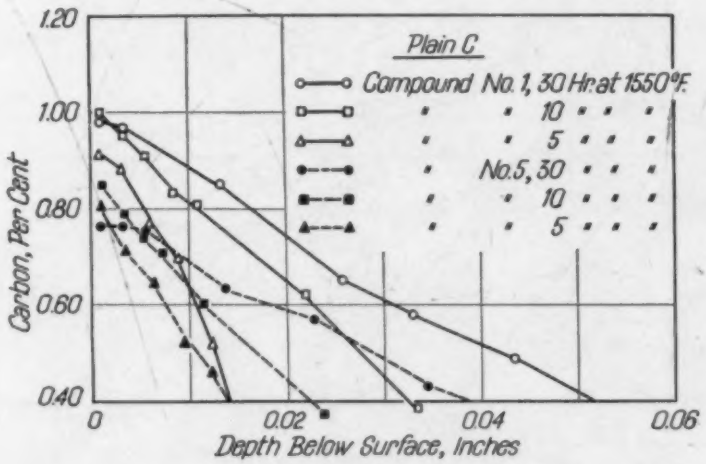


Fig. 16—Effects of Time at 1550 Degrees Fahr. Quenched from Carburizing Temperature.

Effect of Time

Specimens of chromium-molybdenum (A), nickel-chromium and plain carbon steels were again carburized in compounds No. 1 to No. 5 to determine the effect of time. Cycles of 5 and 30 hours were used. Two different carburizing temperatures were used, 1550 and 1700 degrees Fahr. (845 and 925 degrees Cent.), and all speci-

mens were direct quenched from the carburizing box. Results are shown in Figs. 14 to 19.

The results indicate, in general, that for either of the two temperatures used, the longer the time the higher the surface carbon

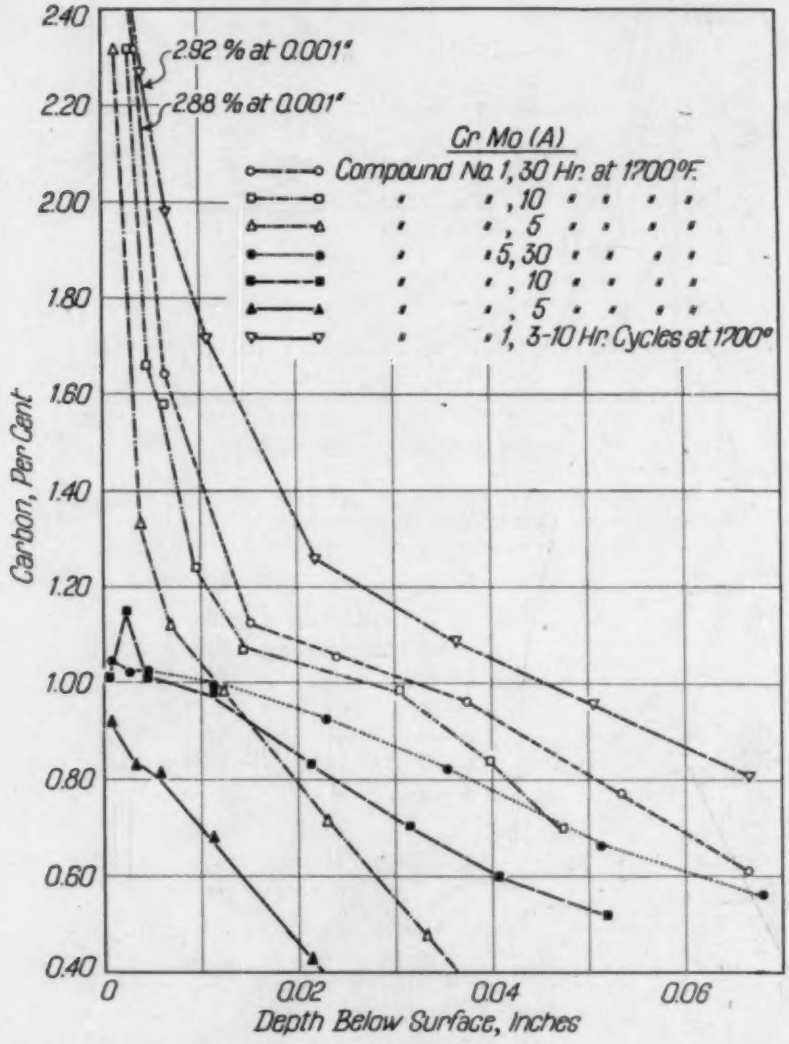


Fig. 17—Effects of Time at 1700 Degrees Fahr. Quenched from Carburizing Temperature.

and the deeper the case. Only two exceptions were found (Figs. 16 and 18) among the specimens carburized in No. 1 compound. The deviation in the plain carbon steel where the 10-hour specimen showed a higher carbon content than the 30-hour specimen is only 0.02 per cent carbon. The deviation in the nickel-chromium steel where the 5-hour test was 0.05 per cent higher in carbon than the 10-hour sample is also a very small one. Of the specimens car-

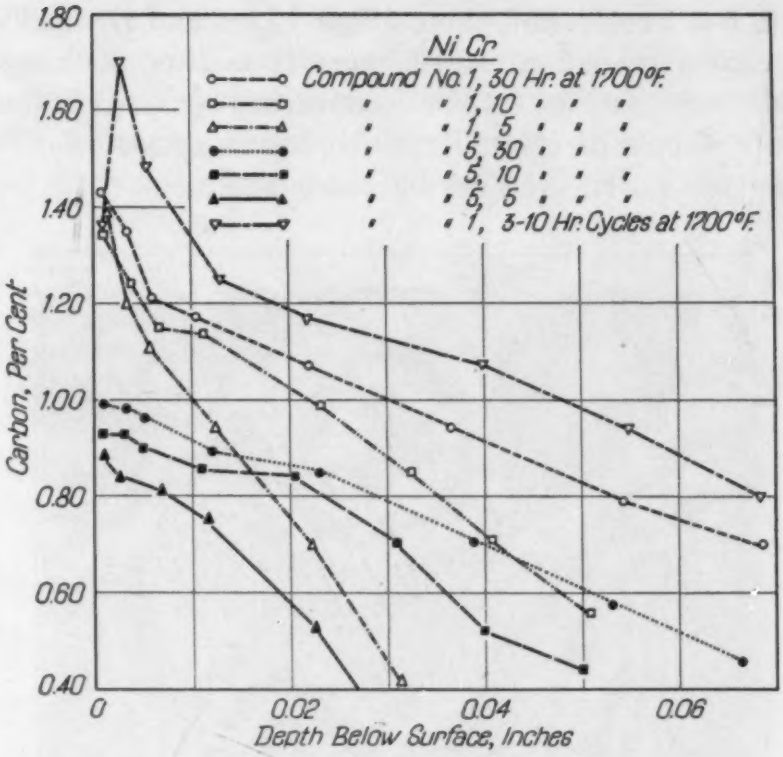


Fig. 18—Effects of Time at 1700 Degrees Fahr. Quenched from Carburizing Temperature.

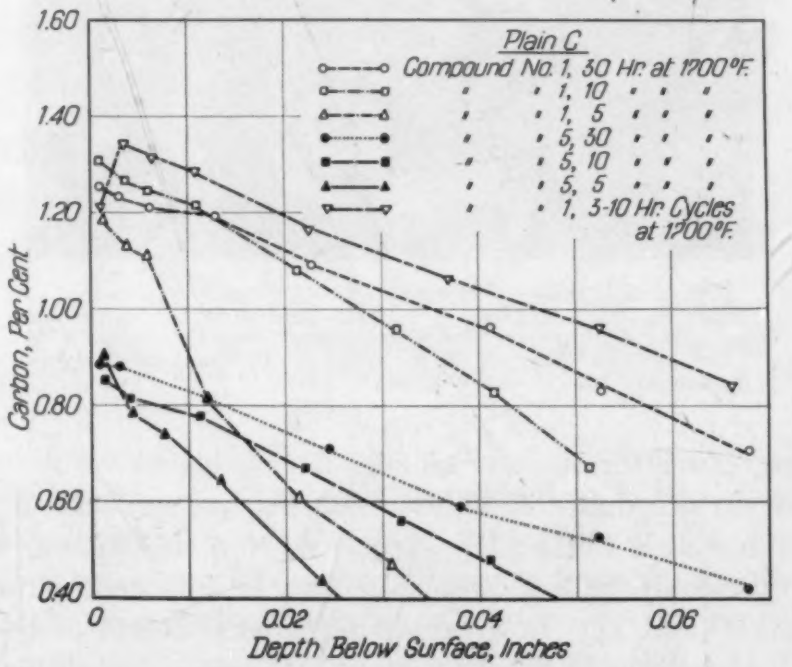


Fig. 19—Effects of Time at 1700 Degrees Fahr. Quenched from Carburizing Temperature.

burized in No. 5 compound, three (Figs. 15, 16 and 17) had a higher surface carbon content after 10 hours than after 30 hours. The maximum discrepancy of 0.12 per cent carbon occurred in the second cut from a sample of chromium-molybdenum (A) steel (Fig. 17). Doubtful points were verified by check analyses.

Fig. 20—Chromium-Molybdenum (A) Steel Carburized 5 Hours in Compound No. 1 at 1700 Degrees Fahr. Quenched from the Carburizing Temperature and Drawn 3 Minutes in Lead at 1200 Degrees Fahr. Top of Photomicrograph was Surface at Time of Carburizing. × 1000.

The reason for 10-hour samples having higher carbon contents near the surface than the 30-hour samples was undoubtedly due to the presence of a thicker low carbon skin on the 30-hour samples. The formation of the low carbon skin may be followed by comparing Figs. 13, 20 and 21. Low carbon skins were found to be present on all the steels carburized for times of longer than 5 hours at a temperature of 1700 degrees Fahr. (925 degrees Cent.) in compound

No. 1. They are believed to have been present on all steels carburized for longer than 5 hours at temperature of 1700 degrees Fahr. (925 degrees Cent.) in compound No. 5 although direct evidence was sometimes lacking because of the absence of excess carbides in quenched specimens. In some instances the depth of the low carbon

Fig. 21—Chromium-Molybdenum (A) Steel Carburized 30 Hours in Compound No. 1 at 1700 Degrees Fahr. Quenched from the Carburizing Temperature and Drawn 3 Minutes in Lead at 1225 Degrees Fahr. Top of Photomicrograph was Surface at Time of Carburizing. × 1000.

skin was sufficient to be apparent in the chemical analyses, in other instances it was not. The tests carburized at 1550 degrees Fahr. (845 degrees Cent.) failed to give any evidence of a low carbon skin either by chemical analyses or by microscopic examination even after the 30-hour cycles. See Fig. 22.

The results indicated that the low carbon skin was produced during the carburizing treatment, that it began to form after approxi-

mately 5 hours at 1700 degrees Fahr. (925 degrees Cent.), and that from there on the depth of this zone increased with time at temperature. Any explanation of the phenomena must take into consideration two observed facts: (1) that the depth of the decarburized zone increased with time, and (2) that both the width of the hyper-

Fig. 22—Chromium-Molybdenum (A) Steel Carburized 30 Hours in Compound No. 1 at 1550 Degrees Fahr. Quenched from the Carburizing Temperature and Drawn 3 Minutes in Lead at 1225 Degrees Fahr. Top of Photomicrograph was Surface at Time of Carburizing. × 1000.

eutectoid zone and the depth of the case continued to increase with time. This precludes simple decarburization as an explanation for the effect.

Figs. 17, 18, 19 also contain the results obtained by carburizing for three 10-hour cycles at 1700 degrees Fahr. (925 degrees Cent.) in compound No. 1. Samples were repacked in new compound after each 10-hour period. The tests were made to determine if loss of

efficiency by the carburizing compound was affecting the depth of carburized case. It was evident that compound No. 1 did lose some of its potency before the end of the 30-hour cycle because in all three instances the case depth of the samples carburized for three 10-hour cycles was greater than the case depth of the samples carburized 30 hours without interruption.

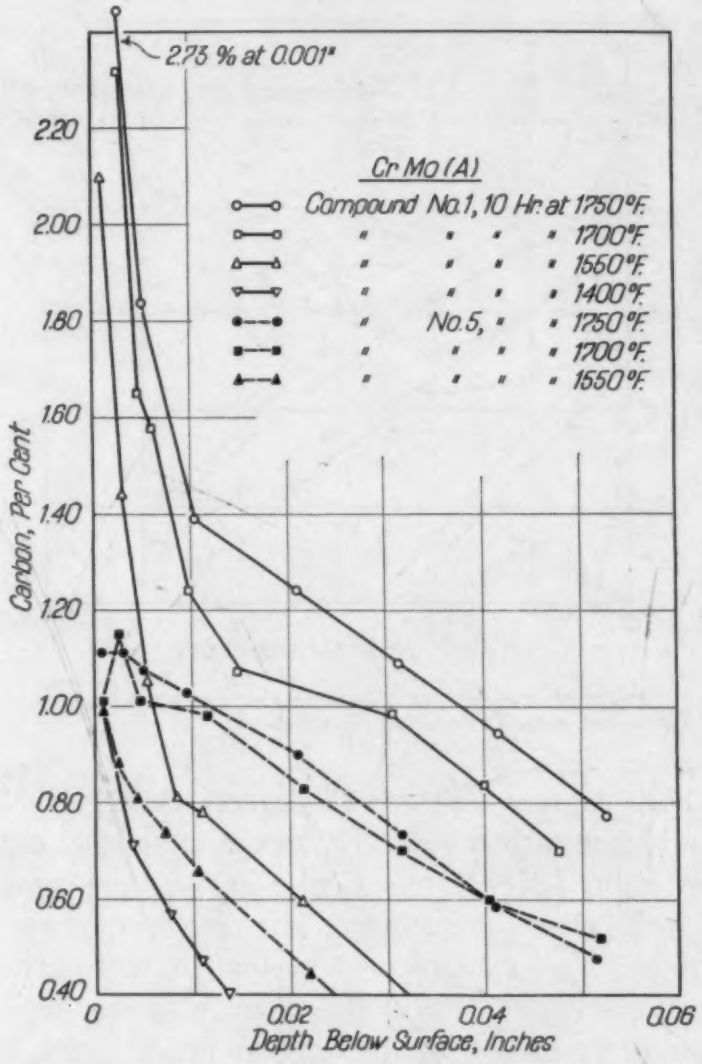


Fig. 23—Effect of Temperature on Case Depth. Quenched from Carburizing Temperature.

Effect of Temperature

Additional samples were carburized at 1750 and 1400 degrees Fahr. (955 and 760 degrees Cent.) and the results cross plotted with the results already shown for temperatures of 1700

and 1550 degrees Fahr. (925 and 845 degrees Cent.) The curves are shown in Figs. 23, 24 and 25.

From the curves it can be seen that for a fixed carburizing time of 10 hours the higher the temperature, the higher was the surface carbon content (disregarding the low carbon skin) and the deeper the case. The chromium-molybdenum (A) steel (Fig. 23) car-

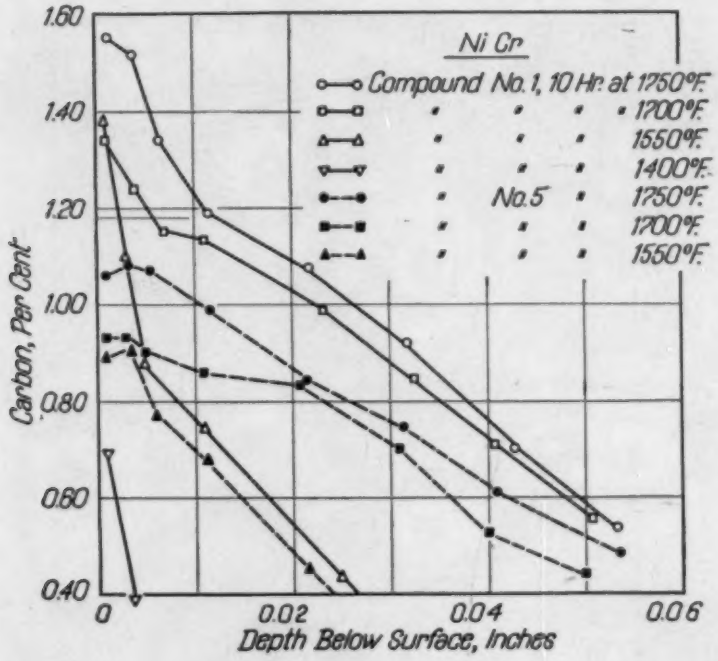


Fig. 24—Effect of Temperature on Case Depth. Quenched from Carburizing Temperature.

burized at 1700 degrees Fahr. (925 degrees Cent.) in No. 5 compound had a higher carbon content than the same steel carburized at 1750 degrees Fahr. (955 degrees Cent.) in the same compound at a point 0.052 inch below the surface. The discrepancy was only 0.04 per cent carbon. Two examples where the curves for two different carburizing temperatures cross at the extreme surface may be noted. These appear to be caused by an increase in the depth of the low carbon skin at higher carburizing temperature.

The carbon analyses of samples taken after carburizing 10 hours at 1400 degrees Fahr. (760 degrees Cent.) in compound No. 5 were all too low to be plotted on the graphs.

Even though the results were compared on the basis of comparable case depth rather than fixed carburizing time there seemed to be little suggestion that the lower carburizing temperatures pro-

duced higher surface carbon. Such a comparison was afforded by Figs. 14 to 19. Samples carburized 30 hours at 1550 degrees Fahr. (845 degrees Cent.) had a comparable case depth to those carburized 5 hours at 1700 degrees Fahr. (925 degrees Cent.). Yet in only two out of six examples did the samples have higher surface carbon after 30 hours at 1550 degrees Fahr. (845 degrees Cent.) than after 5 hours

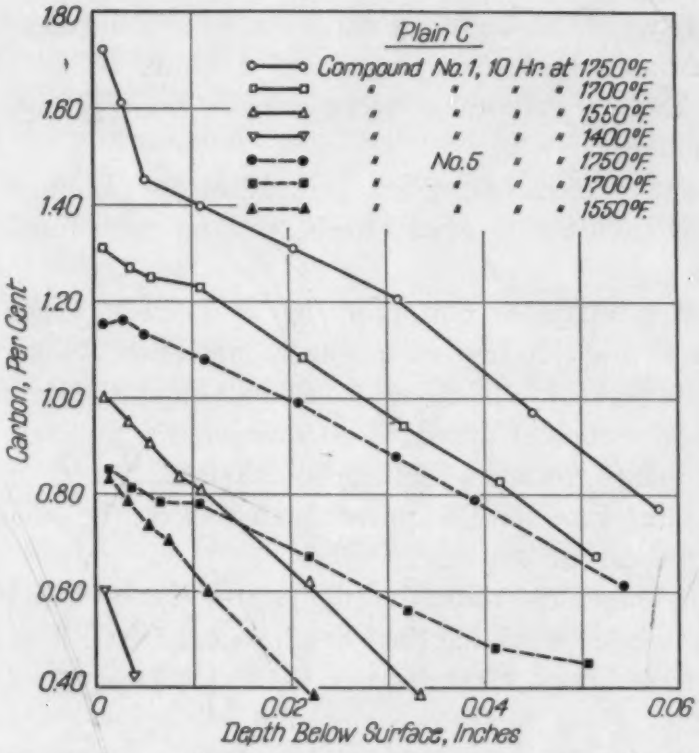


Fig. 25—Effect of Temperature on Case Depth. Quenched from Carburizing Temperature.

at 1700 degrees Fahr. (925 degrees Cent.). And this in spite of the fact that 30 hours at 1550 degrees Fahr. (845 degrees Cent.), in general, produced a slightly deeper case than 5 hours at 1700 degrees Fahr. (925 degrees Cent.). It should be stressed that the results apply only to the specific conditions used for the tests.

The fact that lower carburizing temperatures did not produce cases with higher surface carbon concentration is believed to be in disagreement with widely accepted opinion. For example, on page 1039 of the Metals Handbook (1939 Edition) a graph is presented which shows the surface carbon to be higher the lower the carburizing temperature. Differences in the carburizers employed, and perhaps differences in procedure and even the steels used, may account for the differences in results that were obtained.

Summary

Care should be taken in attempting to apply any portion of the summary to carburizing conditions which do not compare with the conditions employed in this investigation. Specifically, a limited number of certain commercial solid carburizers were tested, eight commercial grades of carburizing steel were tested, a temperature range of 1400 to 1750 degrees Fahr. (760 to 955 degrees Cent.) was used, carburizing cycles varying from 5 hours to 30 hours were used, and cooling conditions were varied from oil quenching to furnace cooling.

1. Alloying elements, when present in the amounts generally found in the carburizing grade steels, had no pronounced effect on case depth.

2. The presence of chromium led to extreme carbon build-up at the surface, when carburized in some compounds. Nickel tended to modify the effect of chromium to some extent. One of the five carburizers investigated appeared to overcome completely the tendency of chromium toward a high carbon surface.

3. Cooling rate had no appreciable effect on the surface carbon content of the carburized case.

4. The longer the time, the higher was the surface carbon and the deeper the case, providing the low carbon skin 0.0003 to 0.001 inch deep after 10 hours at 1700 degrees Fahr. (925 degrees Cent.) was disregarded.

5. For a constant time, the higher the temperature, the higher was the surface carbon and the deeper the case, again providing that the low carbon skin was disregarded.

Acknowledgment

The author wishes to acknowledge his indebtedness to Messrs. R. S. Archer and R. D. Allen for their helpful criticism and suggestions. Special credit is also due W. D. Riddle who performed most of the testing, including the tedious task of recentering the samples in the lathe after carburizing.

Appendix

Procedure

Samples of the various steels were forged into 2.25-inch rounds, cut into 4-inch lengths, and machined to 2 inches in diameter. Many of these 2-inch rounds were later machined to 1.5 inches in diameter and used for another carburizing treatment. Prior to carburizing, the rounds were polished with 180X emery cloth until all cutting tool marks were removed.

Two Nichrome carburizing boxes, one with inside dimensions 9 by 7.5 by 4.75 inches, and the other 8 by 6 by 5.25 inches, were used. Not more than four rounds, each with its cylindrical axis vertical, were packed in a box. One hundred per cent new compound was used in all treatments.

I. Effect of Compound and Alloy Content—The effects of different alloy contents and carburizers were studied by carburizing samples of each grade of steel for 10 hours at 1700 degrees Fahr. (925 degrees Cent.) in each of the five solid carburizing compounds. These specimens were air-cooled in the carburizing box.

II. Effect of Cooling Rate—Specimens of the plain carbon, nickel-chromium and chromium-molybdenum (A) steels were carburized in compounds Nos. 1 and 5 for 10 hours at 1700 degrees Fahr. (925 degrees Cent.) and cooled as follows:

(a.) Oil-quenched from 1700 degrees Fahr. (925 degrees Cent.).

(b.) Furnace-cooled in the box at a rate of approximately 2.5 degrees Fahr. per minute through the critical range. (Total time to cool from 1700 to 1000 degrees Fahr. (975 to 540 degrees Cent.) was 3½ hours.)

(c.) Air-cooled in box.

III. Effect of Time—Specimens of the plain carbon, nickel-chromium, and chromium-molybdenum (A) steels were given the following treatments in compounds Nos. 1 and 5:

(a.) Five hours at 1700 degrees Fahr. (925 degrees Cent.), oil quench.

(b.) Ten hours at 1700 degrees Fahr. (975 degrees Cent.), oil quench.

(c.) Thirty hours at 1700 degrees Fahr. (925 degrees Cent.), oil quench.

(d.) Thirty hours at 1700 degrees Fahr. (925 degrees Cent.) in three 10-hour cycles. After each 10-hour period the samples were oil-quenched, cleaned in carbon tetrachloride, dried, and repacked in fresh compound. (No. 1 compound only was used for this treatment.)

(e.) Five hours at 1550 degrees Fahr. (845 degrees Cent.), oil quench.

(f.) Ten hours at 1550 degrees Fahr. (845 degrees Cent.). oil quench.

(g.) Thirty hours at 1550 degrees Fahr. (845 degrees Cent.), oil quench.

IV. Effect of Temperature—Specimens of the plain carbon, nickel-chromium and chromium-molybdenum (A) steels were carburized in compounds Nos. 1 and 5 as follows:

(a.) Ten hours at 1750 degrees Fahr. (955 degrees Cent.), oil quench.

(b.) Ten hours at 1700 degrees Fahr. (955 degrees Cent.), oil quench.

(c.) Ten hours at 1550 degrees Fahr. (845 degrees Cent.), oil quench.

(d.) Ten hours at 1400 degrees Fahr. (760 degrees Cent.), oil quench.

After carburizing, all quenched specimens were drawn in lead for 3 minutes at 1225 degrees Fahr. (665 degrees Cent.); all furnace-cooled and air-cooled specimens were drawn in a furnace for 3 hours at 1225 degrees Fahr. (665 degrees Cent.) while still in the box.

After drawing specimens were recentered in a lathe to within ± 0.0005 inch (± 0.001 inch in a few cases where warpage occurred). All carbon which had deposited on the surface of the specimens was removed with 180X emery cloth, and the polishing was continued until the surface appeared bright.

Cuts approximately 0.002 inch deep were taken at appropriate positions below the specimen surface and the chips analyzed for carbon by the combustion method. The turnings collected for analysis were confined to a band 2 inches wide at the center of each 4-inch long specimen.

Specimens of which photomicrographs were to be made were plated with 0.001 inch of chromium, then mounted in Bakelite and polished.

DISCUSSION

Written Discussion: By A. S. Jameson, works metallurgist, International Harvester Co., Chicago.

The author's paper is reviewed in light of the writer's own experience and practical experiments in the same field as well as the experience of others.

The paper covers quite a large field dealing with the effect of carburizing compounds, alloy compositions, carburizing time, carburizing temperatures and the effect of the rate of cooling from carburizing on the carbon distribution. It is stated in the introduction that a chromium-molybdenum steel containing about 1 per cent chromium when carburized builds up a high carbon concentration at the surface. This is the view held by the writer and others and is borne out by the author's data. An analysis which has a lower chromium content known as 4119 was developed to minimize this condition. According to the author's data the lower chromium content analysis does not appreciably correct this excessive carbon build-up. He shows that a change in the analysis of the compound can, however, effect a lowering of the surface carbon. By the way, the addition of silicon to the analysis up to 0.80 per cent can also have the same effect. It can also be controlled in gas carburizing by shutting off the main flow of gas and holding at the carburizing temperature, this time being known as diffusion time. Later on in the paper, the author's data show that nickel decreases the carbon content at the surface. This is in accord with established data and experience. It is doubtful that molybdenum would cause a lower surface carbon content. On the contrary, molybdenum tends to increase surface carbon concentration especially in the presence of chromium.

Tests we have made show a relationship as to near surface carbon concentration between five steel compositions to be as follows:

Steel Symbol	Carbon Content (Per Cent) at 0.010 Under Surface	Carbon Content (Per Cent) at 0.005 Under Surface
A-4120	1.27	1.40
A-5120	0.98	1.15
A-3120	0.93	0.99
A-4620	0.88	0.90
A-2317	0.82	0.83

This is using a coke and charcoal compound of the following chemical analysis —barium 7.75, sodium 0.1, calcium 0.44 and iron 0.8, and after carburizing at 1700 degrees Fahr. (925 degrees Cent.) for 8 hours.

The author's data show that the case depth is not affected by the alloy content. This is not in accordance with general experience. Taking the data from the author's Figs. 1 to 8 (inclusive) the case depth measuring to 0.60 per cent carbon for compound No. 1 was:

Steel Symbol	Case Depth (Inches)
4120 (1.06 Mn)	0.046
5120 (0.44 Mn)	0.046
3120	0.046
4119	0.064*
C-1022	0.048
2317	0.045
4620	0.049
4815	0.046

*Interpolated.

We have data showing case depth to 0.50 per cent carbon relationships for five steel compositions to be as follows after carburizing for 8 hours at 1700 degrees Fahr. (925 degrees Cent.):

Steel Symbol	Case Depth (Inches)
A-4120	0.050
A-5120	0.044
A-3120	0.040
A-4620	0.038
A-2317	0.032

These differences require adjustment of carburizing cycles at least between the nickel and chromium-molybdenum steels.

It is known that a decarburized skin is present in steels carburized and slowly cooled but it is assumed that this takes place in cooling from the carburizing temperature and is not present during carburization. Steels therefore quenched from carburizing heat have little or no decarburized zone. Also, it has been noted that the higher the energizer content of the compound the greater will be the decarburized band on slow cooling. It is also noted that steels with a high oxygen content are particularly apt to have decarburized skins often to a depth of 0.010 inch after slow cooling. To a limited degree oxygen may be absorbed at the surface in all commercial carburizing operations. Let it suffice to say that the reactions at the surface of steel are very complex and that the carburizing action can be reversible depending on the compound composition and temperature fluctuations.

The effect of time on the case depth for a nickel-chromium 3120 steel as taken from the author's Fig. 18 would show case depths to 0.55 per cent carbon at varying carburizing times using No. 1 compound as follows:

Time in Hours	Case Depth (Inches)
5	0.028
10	0.051
30	0.088*

*Interpolated.

We have after carburizing at 1700 degrees Fahr. (925 degrees Cent.) in a coke and charcoal compound of the following composition: Barium 4.9, calcium 1.2, and sodium 0.3 per cent:

Time in Hours	Case Depth (Inches)
5	0.034
10	0.051
30	0.078

As to the effect of the carburizing temperature on the case depth to 0.55 per cent in Fig. 24 the author has for a nickel-chromium 3120 steel carburized in compound No. 1 the following data:

Carburizing Temperature Degrees Fahr.	Case Depth (Inches)
1550	0.019
1700	0.048
1750	0.052

We have the following for a similar analysis after carburizing for 16 hours using a coke-charcoal compound analyzing—barium 4.9, calcium 1.2, sodium 0.3 per cent:

Carburizing Temperature Degrees Fahr.	Case Depth (Inches)
1650	0.053
1700	0.070
1750	0.095

Our data are not comparable except to show that the case depth increases as the carburizing temperature is raised. We would like to mention here that the author's data at least for this steel do not agree with ours or others where a difference of only 0.004-inch case depth is obtained between carburizing at 1700 and 1750 degrees Fahr. (925 and 955 degrees Cent.).

The author raises the question as to the carbon concentration for different carburizing temperatures where his data do not agree with the data presented on page 1039 of the A.S.M. METALS HANDBOOK.

He shows for the nickel-chromium steel the carbon contents at 0.010 inch under the surface to be:

Carburizing Temperature Degrees Fahr.	Carbon Content (Per Cent)
1550	0.78
1700	1.15
1750	1.25

Our data after 16 hours of carburization for a coke-charcoal compound containing—barium 4.9, calcium 1.2 and sodium 0.3 per cent show:

Carburizing Temperature Degrees Fahr.	Carbon Content (Per Cent)
1650	1.13
1700	1.22
1750	1.30

The only explanation which could be advanced to account for high carbon concentration at the surface as shown in A.S.M. METALS HANDBOOK is that they represent longer carburizing times. Longer carburizing time means greater case depths and greater case depths mean decreased rate of diffusion from the surface. It is possible under certain conditions that carbide formation at the surface can proceed at a rate greatly in excess of the rate of diffusion. These conditions can be brought about by low carburizing temperature, complex carbide formation such as in chromium-molybdenum steels, and a high carbon content layer between the surface and the low carbon core (deep case).

We wish to say in closing that considering the wide scope of the paper, the author has carried out a thoughtful and well planned series of experiments in the carburizing field.

Written Discussion: By O. W. McMullan, metallurgical department, The Youngstown Sheet and Tube Co., E. Chicago, Ind.

The exact details of the author's procedure in making the three 10-hour tests are not given in the paper but it appears possible that the greater case depth than obtained in the single 30-hour period came from diffusion and additional carburizing that occurred during heating-up time of the two reheats.

Apparently as yet unknown conditions affect the maximum surface content of carburized steel. The writer has observed, at least by appearance under the microscope, higher carbon contents after higher carburizing temperatures as reported by the author, but there also has been considerable evidence to the contrary.

The writer also frequently has observed the low carbon zone with absence of carbides near the surface as reported by the author after carburizing at 1700 degrees Fahr. (925 degrees Cent.). An extreme example of this has been seen in steel carburized in certain gas mixtures that break down to form soot or coke deposits on the surface of the work. Such deposits protect the work from contact with the active carburizing gases and probably create different equilibrium conditions that cause severe decarburization and produce considerable free ferrite in the surface zone of work that has a normal hypereutectoid case underneath. This experience suggests the possibility that a film, perhaps of inactive carbon, builds up on the surface during compound carburizing and that such a film either interferes with whatever function the energizer performs or serves as a shield against the reaction of the active carburizing gases with the steel surface. Both compound composition and carburizing temperature could very well have an influence on the nature of this film as suggested by the differences in appearance of carburized work.

Since the author observed the beginning of low carbon skin formation at the end of 5 hours at 1700 degrees Fahr. (925 degrees Cent.), the writer suggests that additional experiments be made in which samples are removed at the end of 5 hours, carefully cleaned of film by fine emery, then carburized for another 5-hour period, etc., to determine the effect of film removal.

Written Discussion: By G. C. Riegel, chief metallurgist, Caterpillar Tractor Co., Peoria, Ill.

The most outstanding conclusion which the author has reached from the experimental results which he had so carefully conducted in this report, in the writer's opinion, is the refutation of the commonly accepted belief that lower temperature carburizing always produces a higher carbon content in the surface carbon concentration. This particular conclusion offers a controversial subject for others who have contributed to the literature and doubtless will arouse a number of other experimenters to investigate more fully the nature of these phenomena. Some of the evidence which we have accumulated in our Laboratories tends to corroborate the conclusion reached by the author.

We have also found that there is a marked tendency for decarburization to take place, both in solid pack carburization, gas carburization, and the fluid-gas carburization. On three specimens removed in succession from a Homocarb furnace using natural gas as a carburizing medium, the standard specimen removed and analyzed for carbon from the same charge gave the following results:

After 5 hours rapidly cooled from the furnace:		
1st 0.005 inch depth	....Carbon	1.22 Per Cent
2nd 0.005 inch depth	....Carbon	1.11 Per Cent
After 10 hours rapidly cooled from the furnace:		
1st 0.005 inch depth	....Carbon	1.23 Per Cent
2nd 0.005 inch depth	....Carbon	1.17 Per Cent
After 7 hours in cooling retort:		
1st 0.005 inch depth	....Carbon	0.66 Per Cent
2nd 0.005 inch depth	....Carbon	1.02 Per Cent

We have definitely proved that in transferring carburized work at 1700 degrees Fahr. (925 degrees Cent.) through the atmosphere and into a cooling retort, even though the retort is charged with a slight carburizing gas, con-

siderable decarburization takes place. 0.0005 to 0.003 inch of partially decarburized surface is encountered if more than the usual precautions are not exercised.

It has also been our experience that sodium carbonate (soda ash) is required in pack carburizer to the extent of a minimum of 3 per cent if decarburization of the first few thousandths' surface layers is going to be prevented in tightly luted pack carburizing boxes. There has been objection raised to the attack of sodium compounds on the nickel-chromium alloys in pack carburizing, but we have had no short-lived failures from this practice. Other common carbonate energizers are not sufficiently reactive in the lower temperature range to prevent the reversibility of the carburizing equilibrium during the cooling period.

Written Discussion: By H. H. Lurie, chief metallurgist, Cummins Engine Co., Columbus, Ind.

Mr. Manning is to be congratulated on having selected such an interesting and timely subject. Although W.P.B. did not insist on the use of the NE steels 8020, 8620, and 8720 until about the time this paper was completed, it is regrettable that these were not used instead of the S.A.E. 2320 and 4820 grades which now are almost museum items to the average user of steel. And recently the switch to NE 9420 makes even the 8600 series seem like fairly high alloy steels.

In section E, page 13, under "Effect of Alloying Elements", the statement is made that case depth appears to be independent of the alloying elements. This does not bear out the results obtained, unless the discrepancies could be attributed to variations in the heating cycle, or in the heat samples themselves. To speak of total case obtained has not much practical meaning unless the depth of case above 0.60 per cent carbon is used as a measure. Carbon content below 0.60 per cent does not have great wear resistant properties and it is useful to bind the case to the core. Fig. 4, on the same page, shows a favorable effect for chromium-molybdenum (B), in that a case depth of 0.050 inch of 0.60 per cent carbon, or better, is obtained with all compounds except the erratic No. 3. Strange enough plain carbon steel would rank second in uniformity of results (Fig. 5) in the 10-hour, 1700 degrees Fahr. (925 degrees Cent.) heats, air-cooled in the box. However, the average carbon content below 0.020 inch is appreciably higher in the alloy steel chromium-molybdenum (B), Fig. 4.

Nickel appears to have a lesser graphitizing effect, that is, a resistance to the admission of carbon in the austenite, than would normally be supposed. And chromium, as Figs. 1, 2, and 4 show, does certainly affect the carbon content in the depth range of 0.001 to 0.006 inch.

While the paper was presented mainly for its practical application, it would appear that special precautions could have been taken to prevent the loss of surface carbon, or the formation of a decarburized skin, by using a carbon monoxide-rich or oxygen and oxide-free atmosphere for the cooling medium. The variations in case depth seem rather wide for the conditions employed, unless the samples of each steel used were taken from the widely separated parts of the heat. It would be interesting to see the results of duplicated tests on a few of the series to see how reproducible are the given curves.

Oral Discussion

H. B. KNOWLTON:[1] Our studies agree quite well with what the speaker has said. There are several points to which I would like to call attention.

The micrographs show that with chromium-molybdenum steel when the carbon content is in the neighborhood of 2 per cent, the carbides appear in the massive form. They do not form envelopes or networks such as are found in plain carbon steel after carburizing and slow cooling. Our experience has been that when the hypereutectoid carbon appears in the form of massive carbides, variation in the speed of cooling from carburizing makes no difference in the appearance of the structure so far as carbides are concerned. The amount and distribution of the massive carbides is the same for both the direct quenched and the slowly cooled specimens. This seems to indicate that the massive carbides were formed during carburizing by direct combination between iron and the carbon in the carburizing gas, without prior solution of the carbon in the austenite.

On the other hand, in the case of plain carbon steels which are cooled slowly from carburizing, the carbides appear in the form of a network surrounding the original austenite grains, indicating that the carbon was first dissolved in the austenite and then rejected to the grain boundaries during slow cooling.

We would like to know whether the author's experiences have been similar.

With regard to the question as to why a lower carbon content is produced by a long carburizing period, we cannot give a positive answer, but would like to offer a tentative explanation.

In box carburizing both carbon monoxide and carbon dioxide are present in the carburizing box. The chemical energizer in the carburizing compound attempts to produce carbon dioxide which would tend to lower the concentration of carbon in the case. On the other hand the charcoal, coke, or other forms of carbon in the compound attempt to convert all of the carbon dioxide to carbon monoxide which favors the production of a high carbon content in the case. Reaction between carbon monoxide and iron carburizes the iron and produces more carbon dioxide. In order to produce the highest carbon content in the case it is necessary that the atmosphere be kept as nearly 100 per cent carbon monoxide as possible. To accomplish this there must be maximum activity on the part of the carbon particles (charcoal and coke). On long runs there is a tendency for the charcoal and the coke to burn down leaving somewhat less carbon in the box. While we have never seen the analysis of the gases in a carburizing box during different portions of the carburizing run we suggest that there may be less carbon monoxide and more carbon dioxide toward the end of the run.

If this is true, we would expect a lowering of the per cent of carbon which is dissolved in the steel. The decarburizing action might also affect the massive carbides.

As to whether the formation of massive carbides in chromium-molybde-

---

[1]Metallurgist, Gas Power Engineering Department, International Harvester Co., Chicago.

num steel is due to the chromium content, it is interesting to note that Mr. Jameson's slide shows what we have also found, that the carbon content produced by carburizing 4120 steel is higher than that produced in the case of 5120, in spite of the higher chromium content of the latter steel.

We would also like to ask the author a practical question. Does the presence of massive carbides in the case do good or harm? Are there not many cases where such presence of massive carbides may be an advantage?

Author's Reply

The author appreciates the interest of those who have discussed the paper.

It would seem that at least the chief difference between the data presented by Mr. Jameson and the data presented in this paper is concerning the effect of alloying elements on case depth. It would be interesting to know if Mr. Jameson's results were obtained by carburizing the specimens in the same box at the same time so that there could be no doubt about similar carburizing conditions for the steels tested. Incidentally Mr. Jameson's extrapolation for the case depth of the chromium-molybdenum (B) steel is hardly a fair one. Basing the extrapolation on all the points of the carbon distribution curve rather than on the last two points, a better extrapolation would be 0.056 inch or 0.057 inch rather than the 0.064 inch shown by Mr. Jameson. If Mr. Jameson's procedure of determining case depth is carried out for all of the five compounds used and the results averaged for each steel, there still seems to be no suggestion that alloying elements play an important part in case depth. For example, the average case depth of the chromium-molybdenum (A) steel is slightly less than the average case depth of the 1¾ per cent nickel-molybdenum steel; and the average case depth of the nickel-chromium steel is slightly less than the average case depth of the 3½ per cent nickel-molybdenum steel. No significance can be attached to the fact that in some instances the nickel steels are slightly greater in average case depth than the chromium steels. It only indicates that there is no correlation between case depth and the nickel, chromium, and molybdenum content of the steels tested.

The author agrees with Mr. Knowlton in his comments about the massive carbides. Figs. 13, 20, 21, and 22 are of structures that were quenched from the carburizing box. The massive carbides evident in these figures must have been formed during the carburizing cycle and could not have been formed during the very short time taken to cool the samples to room temperature. As Mr. Knowlton pointed out, once these massive carbides are formed during the carburizing cycle, no network is formed even after slow cooling because the additional carbide will precipitate on the already formed particles. Certainly there are many applications where these massive carbides are not a disadvantage. Clash gears are one example pointed out in the paper where the massive carbides caused a great deal of concern among consumers. The presence of massive carbides results in increased brittleness within the case and a consequent lowering of the ability of the part to withstand shock loading. It is essential that clash gears have as great a resistance to shock loading as possible. On the other hand, there are many other applications where resistance to wear

is practically the sole requisite of the part, and under these conditions the presence of massive carbides might even be considered a definite advantage.

Mr. McMullan's question concerning the procedure used in carburizing specimens for three 10-hour cycles is a pertinent one. In making all the tests, each period was counted from the time at which the carburizing furnace reached the specified temperature. The furnace was heated to the temperature to be used for the test before placing the carburizing box in the furnace. It required only about 15 minutes' time after placing the carburizing box in the furnace for the furnace recorder to again reach the specified temperature. There was no simple means of determining just when the inside of the carburizing box reached the temperature but at least it must have been some time after the furnace itself reached the temperature. Therefore the three consecutive 10-hour cycles must represent a somewhat shorter total carburizing time than does the single 30-hour period. Consequently, the procedure used cannot in itself explain the greater case depth found on the specimen carburized for three consecutive 10-hour cycles.

With respect to Mr. Lurie's questions concerning the reproducibility of the procedure employed, the author acknowledges that there is much to be desired. Figs. 10, 11, and 12 served to indicate the degree of reproducibility that could be expected from the tests. Since the cooling rate from the carburizing temperature was found to have no consistent effect on the carbon distribution of the case, the test, though originally made to determine the effect of cooling rate, in fact became an indication of the reproducibility. Several factors, such as lack of complete homogeneity of the carburizing compound, slight variations in sealing of the carburizing box, accuracy in chemical analysis, and variation in chemistry within the steel samples themselves, probably operated to destroy a high degree of reproducibility. However the reproducibility should have been at least as good as any that can be consistently obtained in commercial practice and appears to be sufficiently good to justify the conclusions that were drawn.

Mr. Lurie implies in his discussion that the decarburized skin was caused by the method used in cooling the specimens from the carburizing temperature. This cannot have been the case as evidenced by the lack of any consistent trend in Figs. 10, 11, and 12. Furthermore, had this decarburized skin been formed during cooling, the samples quenched after 5 hours at the carburizing temperature would be expected to show the same amount of decarburized skin as the samples quenched after either 10 or 30 hours at the carburizing temperature. As it was, samples carburized 5 hours at 1700 degrees Fahr. (925 degrees Cent.) and quenched did not have a decarburized skin, while those carburized 10 hours at 1700 degrees Fahr. (925 degrees Cent.) and quenched had a very thin one, and those carburized 30 hours at 1700 degrees Fahr. (925 degrees Cent.) and quenched had a much thicker one.

A QUANTITATIVE STUDY OF AUSTENITE TRANSFORMATION

By R. A. Flinn, Earnshaw Cook and J. A. Fellows

Abstract

Dilatometric apparatus for the determination of the rate and volume of austenite transformation at all temperatures is described. The salient features of rapid quenching rate and complete accounting for all length changes permit a quantitative interpretation of the precipitation of primary ferrite and of cementite, and the formation of pearlitic, acicular and martensitic structures.

From the dilation curves, summary graphs are prepared to show a method for the location of the "M" point, the progression of martensite formation with carbon content and temperature, and the complete "S" curve.

The data emphasize the narrow temperature range for isothermal annealing of highly alloyed steels, the sluggishness of the later portions of acicular transformation, the instantaneous nature, degree, and high volume change of the martensite reaction.

Review of Literature

SINCE the original illuminating researches of Bain and Davenport (1)[1], in 1930, a number of investigations have employed the isothermal method to analyze the austenite transformation characteristics of steels and cast irons of both simple and complex chemistry (2-8). These data indicate that any particular austenite will transform in one or more of three distinct modes:

1. The transformation to pearlite, to ferrite and pearlite, or to cementite and pearlite, is usually associated with temperatures above 1000 degrees Fahr. (540 degrees Cent.).
2. The intermediate transformation to acicular structures, which occurs below 1000 degrees Fahr. (540 degrees Cent.).
3. The transformation to martensite, which begins at the "M" point and continues as lower temperatures are reached.

[1]The figures appearing in parentheses refer to the bibliography appended to this paper.

Of the authors, R. A. Flinn is assistant metallurgist, Earnshaw Cook is chief metallurgist, and J. A. Fellows is assistant chief metallurgist, The American Brake Shoe and Foundry Co., Mahwah, N. J. Manuscript received November 16, 1942.

With minor variations, the hardness of the products formed for each mode increases in the order named, i.e., inversely with the temperature of reaction. The rates of transformation to pearlite and to intermediate structures are importantly affected by the chemistry of the particular steel or cast iron, because these reactions involve nucleation and growth. Transformation to martensite, however, is substantially instantaneous as the temperature is decreased below the "M" point. The "M" point temperature and the range of martensite formation are, nevertheless, influenced by the analysis of the alloy and may lie between 800 degrees Fahr. (425 degrees Cent.) and sub-zero temperatures.

After a critical examination of the many devices which have been used to observe the progress of transformation, the dilatometer, supplemented by the microscope, seemed to offer the most valuable and comprehensive technique because of simplicity, accuracy, objectivity of observations, and comparative rapidity of determinations. As early as 1919, Chevenard (11) recognized these advantages and constructed a differential dilatometer for the study of transformation during continuous cooling. Bain (1) applied a quenching dilatometer to the investigation of the intermediate and martensitic reactions because of the difficulty of evaluating the transformation rates of these fine structures with the microscope. Oakley and Oesterle (12) have described a dilatometer, designed for a heavy tubular specimen (2 inches long by 0.375 inch I.D. by 0.5 inch O.D.) held in compression. Griffiths, Pfeil and Allen (8) have developed an instrument for a 0.110-inch diameter compression specimen shortened to 1.14 inches to prevent buckling. Parke and Herzig (4) report an instrument with an optical lever for use in the intermediate range, with specimens of 0.017 or 0.034 inch thickness (held in tension) to provide accelerated cooling.

Dilatometric Equipment

In designing an instrument for the study of austenite transformation by all three reactions, it was important that a complete register of all length changes during each test should be possible. Where metallographic specimens are used for determining austenite transformation rates, an unsatisfactory quench will usually be revealed by the presence of spurious micro-constituents, while for the dilatometric technique, only changes in length can be observed. If these

changes are correlated with a base point, such as the length at austenitizing temperature, a check of non-isothermal transformation occurring during quenching is available.

The Dilatometer Proper

The instrument of Figs. 1 and 2 provides a complete record of specimen length variations from time of insertion to time of removal

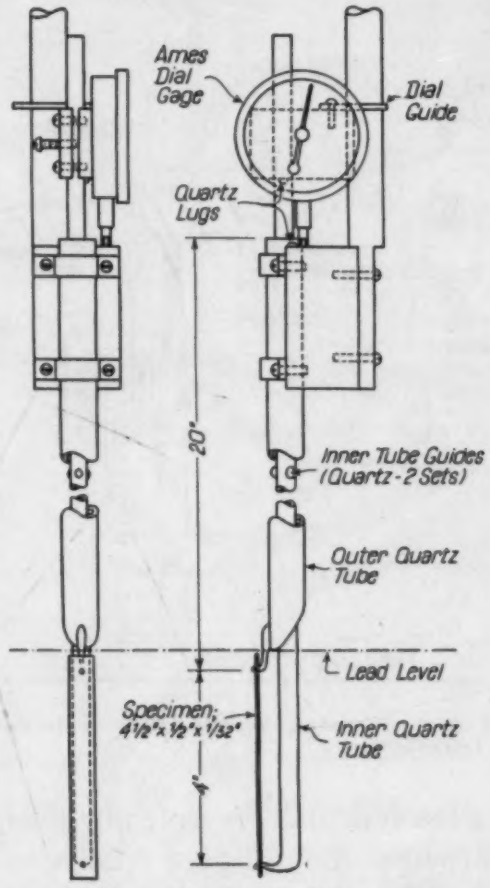


Fig. 1—Details of Dilatometer Specimen Mount and Gage Attachment.

from a lead bath. It also permits change of baths, intermittent cooling to room temperature or below, and general flexibility of operation.

With reference to Fig. 1, an outer quartz tube is held rigidly from a fixed vertical steel shaft by means of a V-block. The specimen is suspended from a quartz hook at the base of this tube, and the inner quartz tube is in turn supported by a hook suspension through the bottom hole in the specimen, maintaining the latter

always in tension. An Ames dial gage (Model 9900, 0.100 inch travel, scale divisions in 0.0001 inch) is clamped to the inner tube. A sheet of thin folded emery paper between the tube and clamp prevents cracking. In operation, dimensional changes of the specimen move the inner tube, which raises or lowers the dial gage relative to the upper surface of the V-block. A light spring maintains the pressure of the dial gage spindle point against the block.

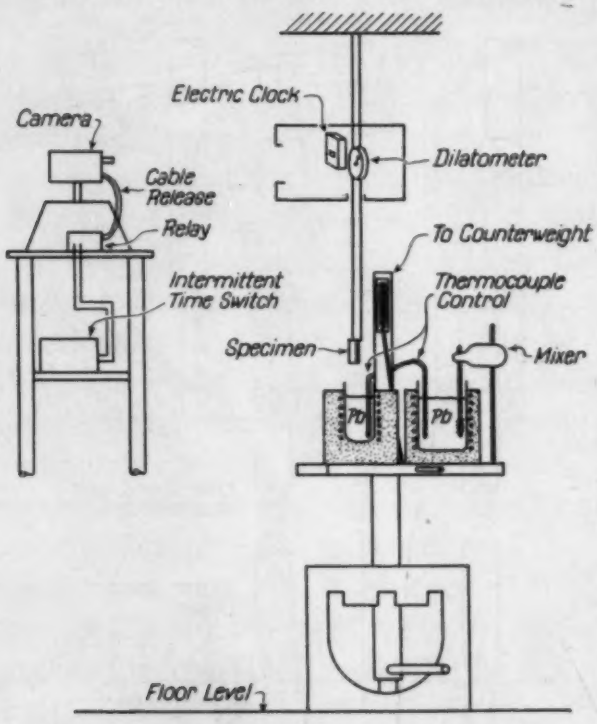


Fig. 2—Schematic Representation of Dilatometer Equipment.

Any slippage in the system is prevented by the quartz lugs which obviate movement of either the dial gage relative to the inner quartz tube or of the outer tube with respect to the V-block. The quartz guides are used for two reasons: (a) to compensate for a generous clearance between the tubes in the lead bath, thus preventing entrapment of lead pellets, charcoal, etc.; and (b) to provide a free, sliding fit between the tubes. The U-shaped dial guide prevents lateral displacement of the gage spindle.

The specimens are 4.5 inches by 0.5 inch by "t" inches. The thickness "t" may be as little at 0.010 inch. Since the tension method of gripping minimizes bowing, thinness of the specimen is limited only by the tendency to creep at the austenitizing tempera-

ture. The specimen section, and its relative isolation from the heated dilatometer parts by means of the hooks, provides a cooling rate which is sufficiently rapid to avoid transformation on quenching to bath temperatures above the "M" point in all the steels investigated.

A specimen of "18-8" steel was quenched from 1650 to 200 degrees Fahr. (900 to 95 degrees Cent.) to provide a blank determination of cooling rate for "t" equal to $\frac{1}{32}$ inch, as shown in Fig.

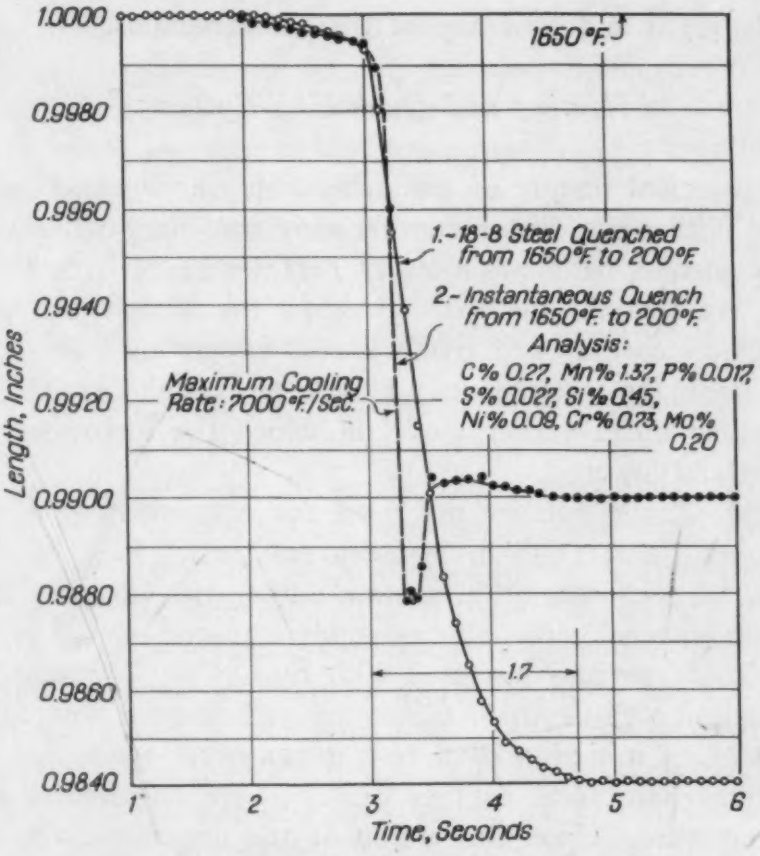


Fig. 3—Cooling Rates Obtainable with Dilatometer.

3, Curve 1. Because of the poorer conductivity of "18-8", this rate is lower than for low alloy steels. The time at which the bottom of the specimen entered the quenching bath is evident from the abrupt change in slope of the graph at 3 seconds. Although the curve indicates that the sample reached bath temperature 1.7 seconds later, over 90 per cent of the contraction and, therefore, of the temperature drop occurs in less than 1.0 second. The quenching rate of any particular segment is faster than indicated by these figures, because 0.4 to 0.5 second was required for completion of immersion.

For Curve 2, Fig. 3, the initial transfer of the dilatometer was to a submerged air chamber, which was then opened to provide an instantaneous lead quench as described later. A maximum cooling rate of 7000 degrees Fahr. (3890 degrees Cent.) per second was obtained. This quantity is more representative of the cooling rate of individual segments of the specimen with the usual technique than the time reported.

The rate of free fall of the dial gage spindle was found to be sufficiently rapid to follow any of these contractions.

Heating and Quenching System

The principal feature of the austenitizing and quenching system illustrated is that the dilatometer remains stationary while the heating and quenching baths are moved. This departure from the apparently less cumbersome method of moving the dilatometer facilitates photographic recording and avoids impact effects upon the hot specimen and upon the instruments. This latter variable caused considerable trouble in an earlier model in which the instrument proper was raised and lowered.

In Fig. 2, the smaller pot used for austenitizing at 1550 to 1650 degrees Fahr. (845 to 900 degrees Cent.) is a 26 per cent chromium:12 per cent nickel casting fitted with an alundum tube wound with chromel wire. This assembly is placed in a 12-inch diameter steel container and insulated with Sil-O-Cel. The receptacle is filled with lead to a depth of 6.5 inches and covered with fine charcoal. Preceding initiation of a test, a controller maintains the pot at 10 degrees Fahr. (5.6 degrees Cent.) above the desired austenitizing temperature. Upon immersion of the apparatus, the sensible heat absorbed by the submerged portion of the dilatometer lowers the temperature of the lead within 30 seconds to the control point which is then corrected. The specimen is maintained at temperature ($\pm$ 3 degrees Fahr.) for 10 minutes.

The quenching receptacle contains approximately 80 pounds of lead for experiments above 700 degrees Fahr. (370 degrees Cent.) and of Wood's metal for lower temperatures. This pot is constructed of low carbon steel, insulated with asbestos paper from a chromel heating element. Temperature is controlled to $\pm$ 2 degrees Fahr. The electric stirrer is operated at high speed from the beginning of the quench until the specimen reaches bath temperature.

Quenching is performed by swinging the stage along a U-shaped course guided by a template provided for the purpose. The elapsed time for the entire operation is about 1.5 seconds. Actual immersion of the specimen accounts for 0.4 second of this time. When transformation is apparently complete or when the time for completion would be inordinately long, the bath is lowered from the dilatometer and the specimen allowed to cool. Room temperature is accurately attained by dipping in alcohol and specimen length is measured. The assembly is then reimmersed in the quenching bath and the final length recorded. Any increase in length occasioned by the intervening quench to room temperature is indicative of austenite previously untransformed at bath temperature. This practice for low alloy steels has been found to compare accurately with uninterrupted isothermal transformations which can be completed within reasonable periods.

Recording, Plotting, Calculating

The dial gage and electric timer (0.1-second intervals to 10,000 seconds) are photographed with a magazine type motion picture camera at film speeds of 30 or 50 frames per second. The first 5 seconds of each experiment are recorded by continuous operation of the camera and thereafter single frame exposures are made at closely spaced logarithmic intervals. When these intervals exceed 30 seconds, a timer-relay system is connected for automatic operation. The exposed film is removed from the magazine, developed for maximum contrast, and projected for recording. The readings of the various graphs have been converted to a standard condition where all dilation curves are inaugurated at a length of 1 inch for a temperature of 1650 degrees Fahr. (900 degrees Cent.). This procedure facilitates calculations for comparing data.

Dilatometer Characteristics

The sensitivity of the dial gage is adequate for the accuracy of the experiments as the gage length may be recorded to 1 part in 400,000 or 0.00025 per cent. The over-all precision of the apparatus is within 2 per cent as demonstrated by duplicate runs, Fig. 10, Curves 2 and 3. The absolute accuracy is only slightly less since thermocouples are calibrated periodically. The continuity of data is satisfactory and their reproducibility is excellent.

The Volumetric Characteristics of Austenite Transformation

The dilatometric manifestations of the three modes of austenite transformation will now be described. While a portion of the data duplicates material which has been published previously, it has been included to illustrate the complete survey of austenite transformation provided by the dilatometer.

Dilation Curves 1300 to 70 Degrees Fahr. (705 to 21 Degrees Cent.) Inclusive

1. High Temperature Transformations (1300 to 1000 Degrees Fahr.)—Austenite to Ferrite + Pearlite, or Cementite + Pearlite.

The high temperature transformations appear in the dilation curves of Fig. 4. The behavior may, in general, be divided into five stages:

(a) Austenite contraction from 1650 degrees Fahr. (900 degrees Cent.) to bath temperature.
(b) Period of incubation with no appreciable length change.
(c) Austenite transformation to hypo- or hypereutectoid constituents; i.e., free ferrite or primary cementite.
(d) Austenite to pearlite reaction.
(e) Period of no further change: transformation complete.

In the first curve of Fig. 4 (1300 degrees Fahr. transformation of 0.30 per cent carbon alloy steel) stages "a" and "b" are evident, but at 180,000 seconds, before completion of ferrite formation, "c", the specimen was quenched in alcohol (80 degrees Fahr.) and then returned to temperature. The increase in length thus produced may be considered a close approximation of the eventual isothermal length change which would have occurred with sufficient time at temperature. This hypothesis has been established by interrupting more rapid transformations and comparing the length for the composite products with the length for isothermal reaction. The more voluminous product formed on quenching is tempered on reheating. Fig. 5 is representative of the composite microstructure.

In the second curve of Fig. 4 (at 1250 degrees Fahr.) all five stages are evident. The change in slope of the dilation curve upon the initiation of the pearlite reaction, stage "d", is characteristic of many hypoeutectoid steels examined at this laboratory and is caused by the more rapid reaction rate accompanying pearlite formation.

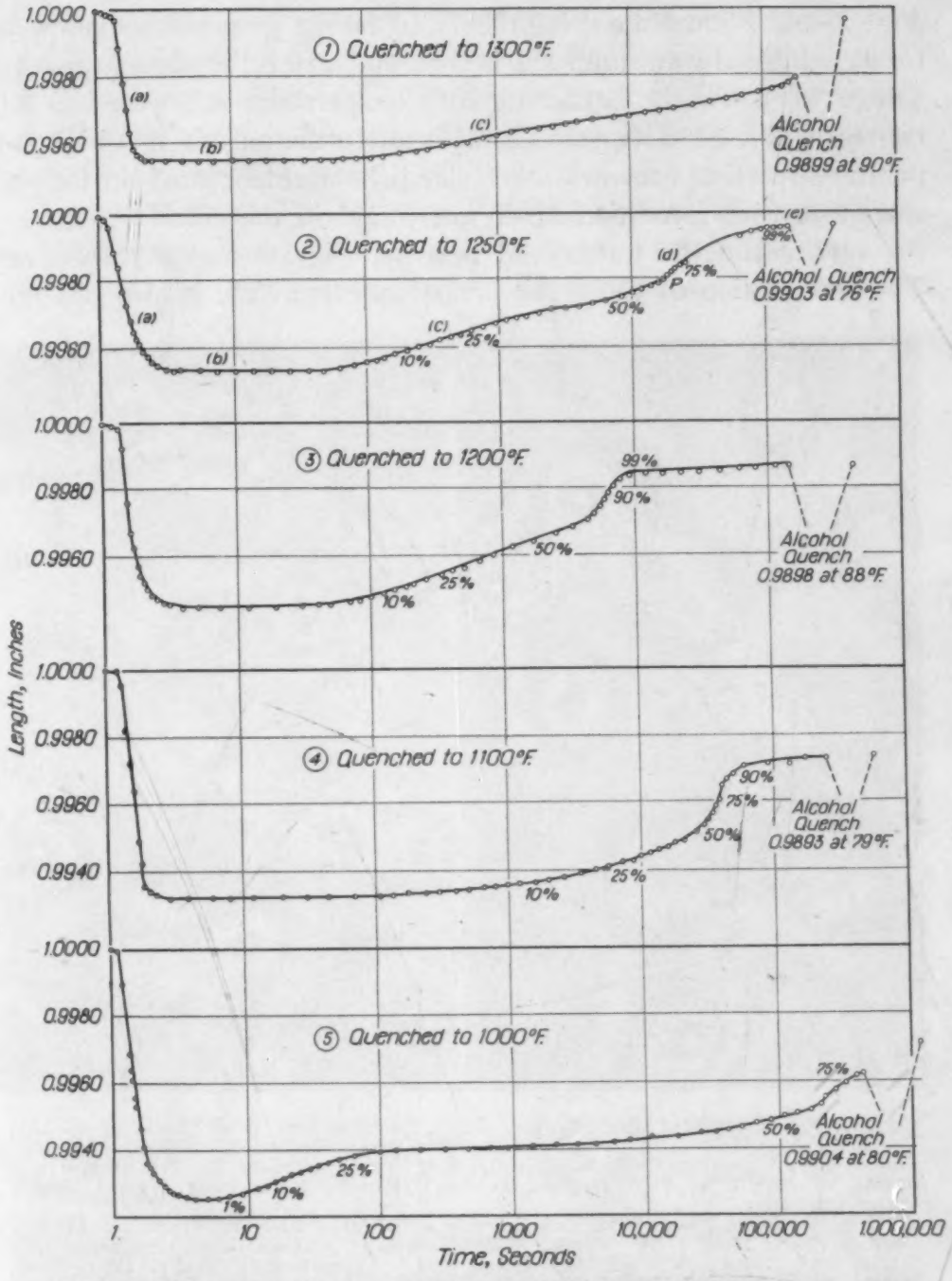


Fig. 4—Dilatometer Curves. Quenches from 1650 to 1300-1000 Degrees Fahr. Inclusive. Analysis of Steel: Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33.

Curves 3, 4, 5, for temperatures of 1200, 1100 and 1000 degrees Fahr. (650, 595 and 540 degrees Cent.) respectively, exhibit the same general habits of transformation as Curve 2. Curve 3 demonstrates that transformation occurs in the shortest time, near 1200 degrees Fahr. (650 degrees Cent.), for this range of temperatures.

Figs. 6 and 7 illustrate the ferrite, and ferrite + pearlite microstructures, which are encountered before and after the change in slope (stage "d"). As the quenching bath temperature is lowered to 1000 degrees Fahr. (540 degrees Cent.) transformation via this high temperature reaction becomes more sluggish. Evidences of an incipient change in transformation mode are found in the acicular nature of the ferrite and the unresolved pearlite, Figs. 8 and 9 respectively. The acceleration of the ferrite reaction, stage "c", is also observed.

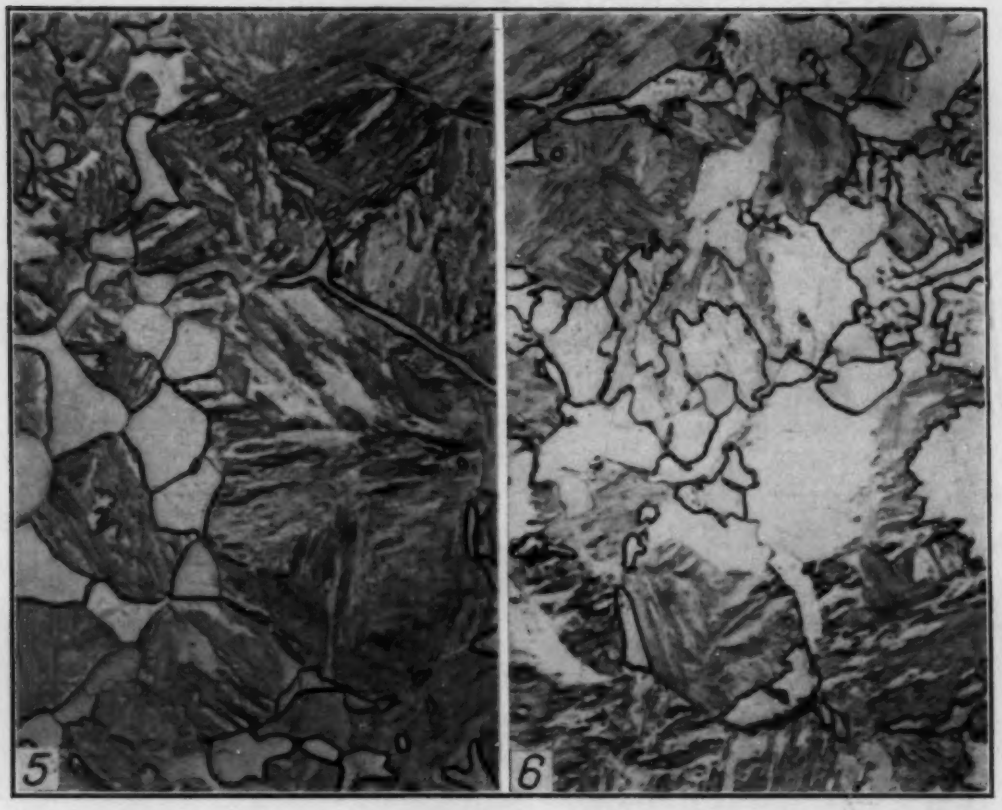


Fig. 5—Austenitized 1650 Degrees Fahr., 10 Minutes. Lead-Quenched to 1300 Degrees Fahr., Held 3500 Seconds, Water-Quenched. Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33. Approximately 25 Per Cent Transformed to Ferrite at 1300 Degrees Fahr., Martensite Background Formed During Water Quench.

Fig. 6—Austenitized 1650 Degrees Fahr., 10 Minutes. Lead-Quenched to 1200 Degrees Fahr., Held 1750 Seconds, Water-Quenched. Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33. Approximately 50 Per Cent Transformed to Ferrite at 1200 Degrees Fahr., Martensite Background Formed During Water Quench. Etch Shows Apparent Gradients in Martensite. Both Specimens Etched in 1 Per Cent Nital, 10 Seconds. × 1000.

The high temperature transformation of hypereutectoid material is illustrated in Curve 1, Fig. 10, at 1300 degrees Fahr. (705 degrees Cent.). After the periods of austenite contraction and incubation, a contraction is observed from carbide precipitation, stage "c". The pearlite reaction then proceeds as discussed previously,

stage "d". With the completed transformation, the primary carbides appear in isolated masses and outlining the pearlite grain boundaries, Figs. 11A and 11B. At 1200 degrees Fahr. (650 degrees Cent.) free cementite and pearlite separation (stages "c" and "d") are not readily differentiated because of the rapid rate of transformation.

2. *Intermediate Temperature Transformations*—Between 1100 and 900 degrees Fahr. (595 and 480 degrees Cent.), the transformation loses the high temperature characteristics described above,

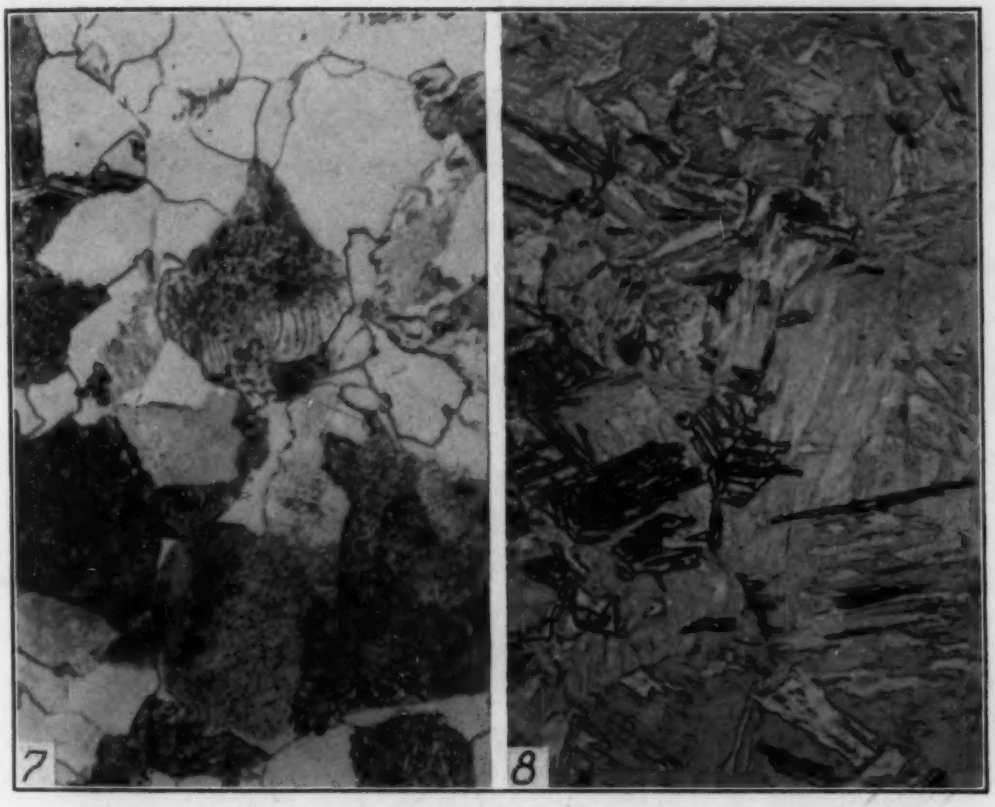


Fig. 7—Austenitized 1650 Degrees Fahr., 10 Minutes. Lead-Quenched to 1200 Degrees Fahr., Held 5200 Seconds, Water-Quenched. Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33. Approximately 75 Per Cent Transformed to Ferrite Plus Pearlite. 10 Per Cent Light Etching Martensite Formed in Water Quench.

Fig. 8—Austenitized 1650 Degrees Fahr., 10 Minutes. Lead-Quenched to 1000 Degrees Fahr., Held 42 Seconds, Water-Quenched. Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33. Approximately 25 Per Cent Transformed to Acicular Ferrite, Martensite Background Formed on Water Quench. Both Specimens Etched in 1 Per Cent Nital, 10 Seconds. × 1000.

and changes to the intermediate mode. The most important differences are the disappearance of pearlite as a reaction product, a change in shape of the ferrite from equiaxed to acicular grains, and decrease in purity of the ferrite, i.e., increased carbon entrapment, first as the amount transformed increases, second as the tempera-

ture of transformation is lowered (7, 6). The following stages may be recognized in the dilation curves for the intermediate transformation:

a. Austenite contraction.
b. Period of incubation.
c. *Rapid* transformation to acicular structures.
d. *Very slow* transformation to acicular structures.
e. Period of no change: transformation complete. With high carbon materials some contraction by isothermal tempering is obtained.

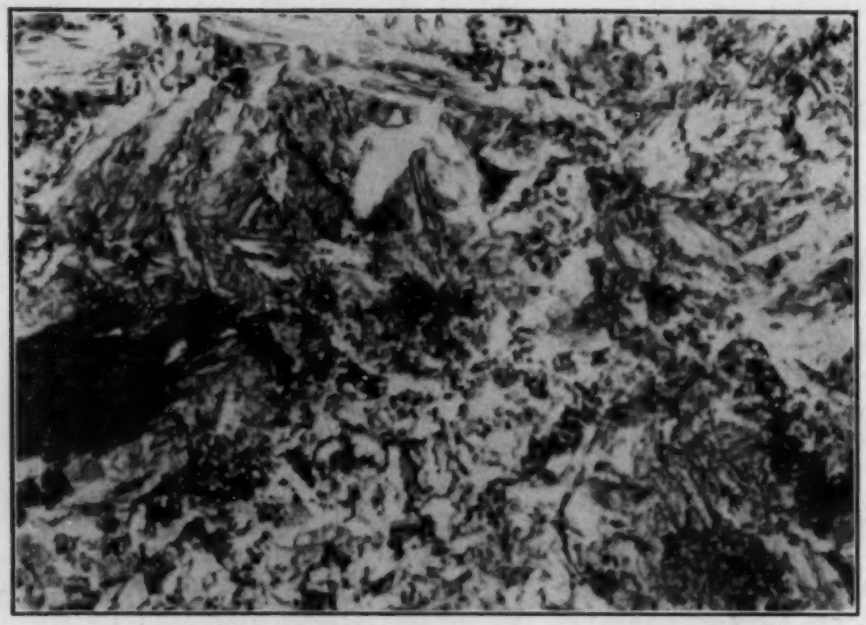


Fig. 9—Austenitized 1650 Degrees Fahr., 10 Minutes. Lead-Quenched to 1000 Degrees Fahr., Held 420,000 Seconds, Alcohol-Quenched. Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33. Approximately 90 Per Cent Transformed to Ferrite Carbide Aggregate. White Patches Are Coalesced Ferrite Needles c.f. Fig. 10. Dark Areas Are Extremely Fine Pearlite. Gray Background is Martensite Formed on Alcohol Quench. Etched in 1 Per Cent Nital, 10 Seconds. × 1000.

In Fig. 12, at 900 degrees Fahr. (480 degrees Cent.), stage "d" is extremely slow; extrapolation of the dilation curve shows that almost a year would be necessary to complete the reaction. Two reasons may be offered for this behavior: First, 900 degrees Fahr. (480 degrees Cent.) is near the maximum temperature for the acicular mode of transformation and, therefore, the driving force is low. Secondly, the separation of a transformation product, which is lower in carbon than the austenite matrix, tends progressively to stabilize the austenite by its enrichment with carbon and perhaps with other

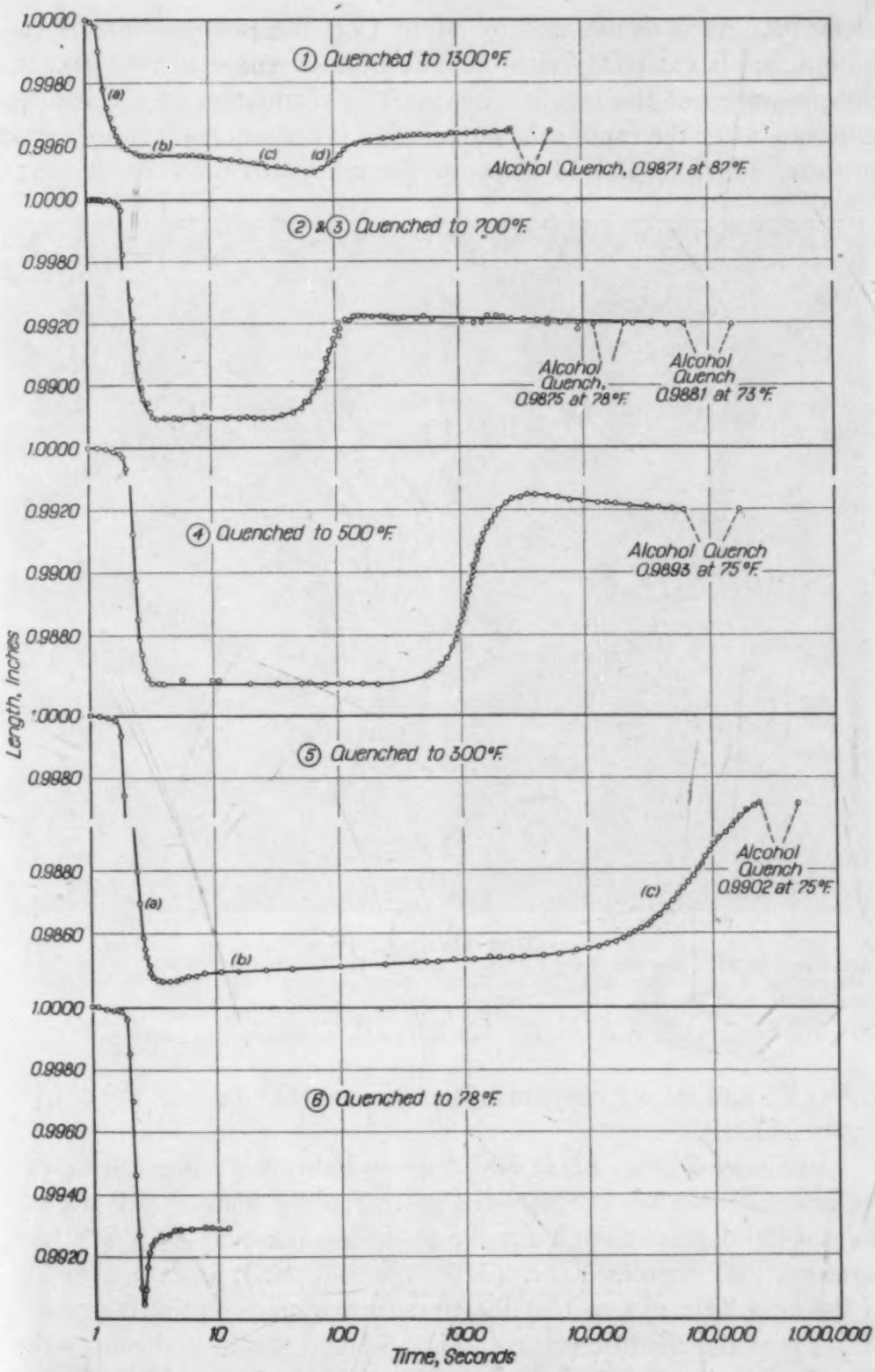
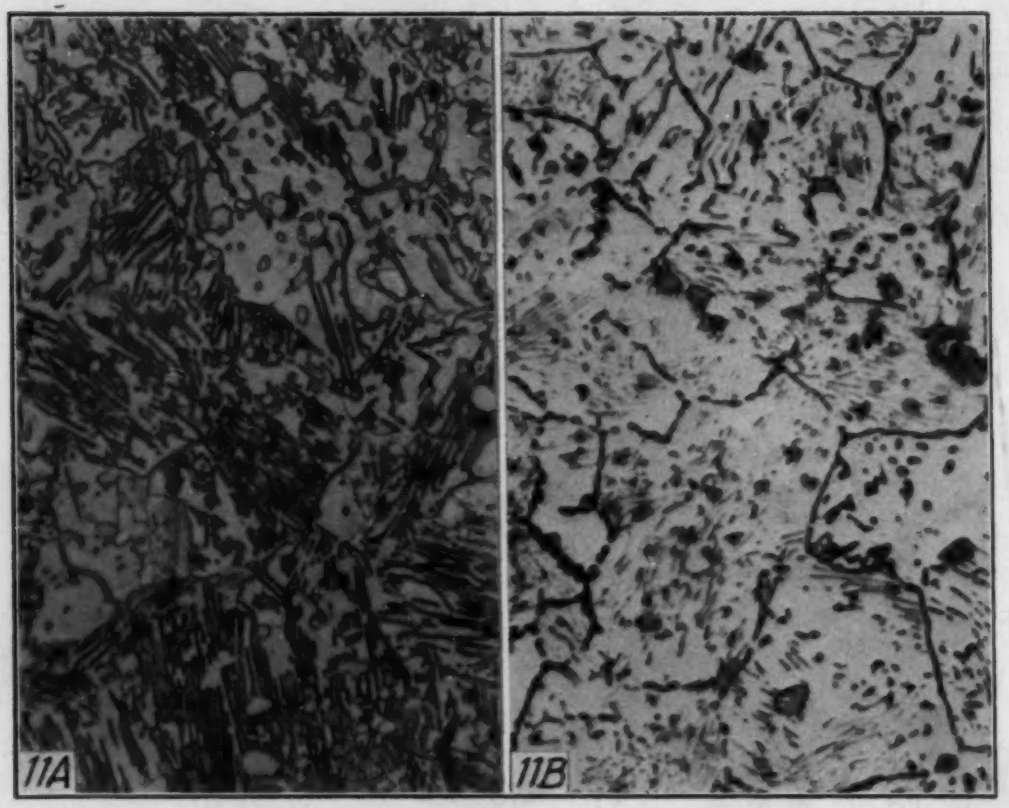


Fig. 10—Dilatometer Curves. Carbon Tool Steel Quenched: 1650 to 1300 Degrees Fahr. (Curve 1). 1600 to 700-78 Degrees Fahr. Inclusive (Curves 2 to 6). Analysis of Steel: Carbon 1.04, Manganese 0.29, Silicon 0.17, Nickel 0.05, Chromium 0.04.

elements. As demonstrated by Mehl (9), the precipitation of carbide nuclei is extremely slow at intermediate temperatures; viz., the disappearance of the pearlitic mode. Transformation of the residual austenite after the rapid acicular reaction is consequently a protracted process. Figs. 13 and 14 illustrate the microstructures for approxi-



Figs. 11A and 11B—Austenitized at 1600 Degrees Fahr., 10 Minutes. Lead-Quenched to 1300 Degrees Fahr., Held 5200 Seconds, Alcohol-Quenched. Carbon 1.04, Manganese 0.29, Silicon 0.17, Nickel 0.05, Chromium 0.04. Completely Transformed to Carbide Plus Pearlite.
Fig. 11A—Etched in 1 Per Cent Nital, 10 Seconds. × 1000.
Fig. 11B—Etched With Alkaline Sodium Picrate, 5 Minutes. × 1000.

mately 25 and 90 per cent transformation at 900 degrees Fahr. (480 degrees Cent.).

In Curve 2, Fig. 12, at 700 degrees Fahr. (370 degrees Cent.), the acicular reaction is completed more rapidly than at 900 degrees Fahr. (480 degrees Cent.) for the following reasons: (A) The temperature, 700 degrees Fahr. (370 degrees Cent.), is in the middle of the acicular range so that the transformation potential is greater; (B) From the microstructures, Figs. 15 and 16, it is demonstrable that the acicular products are darker etching than at 900 degrees Fahr. (480 degrees Cent.), a result attributed to increased carbon

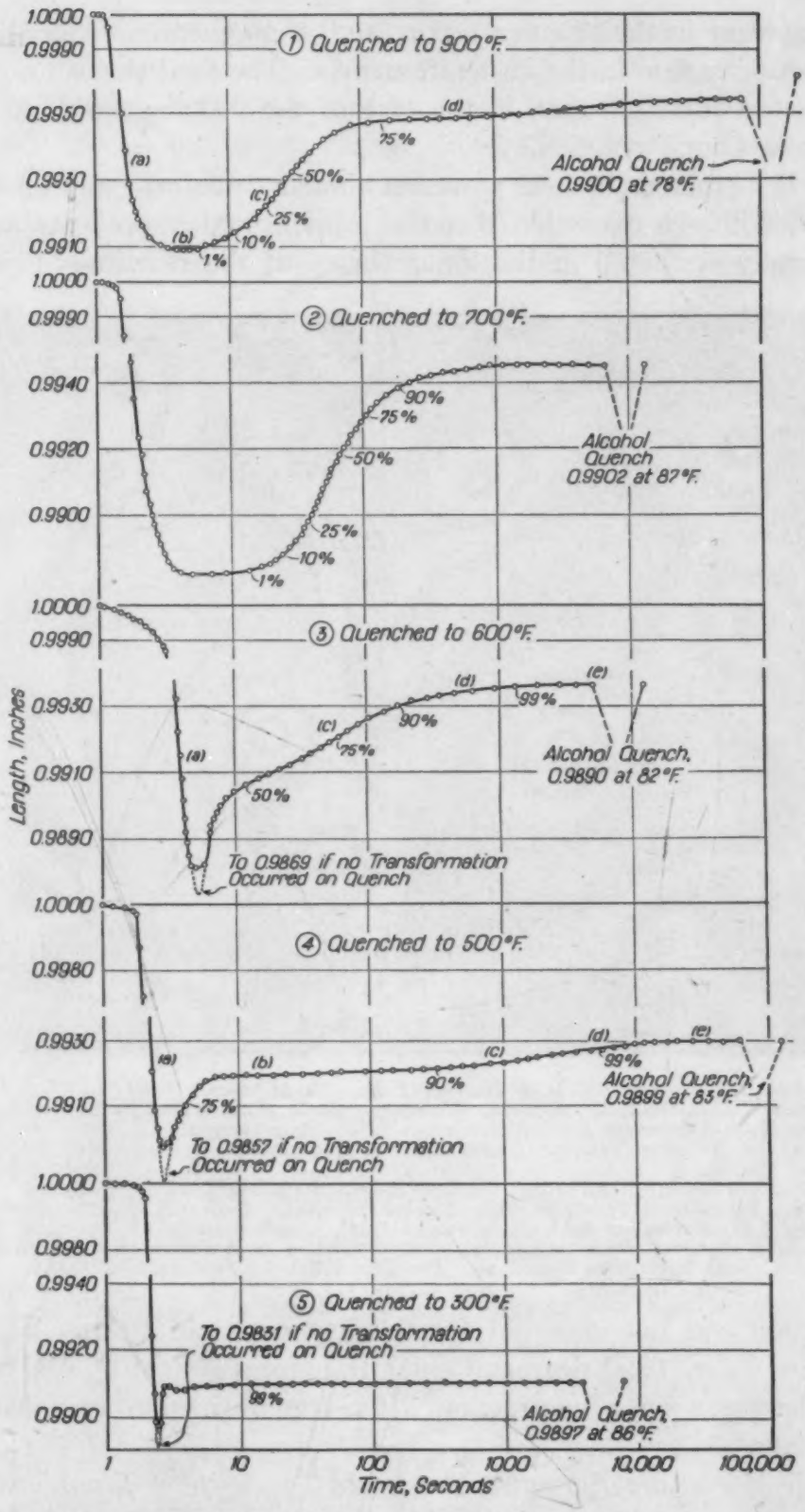


Fig. 12—Dilatometer Curves. Quenches from 1650 to 900-300 Degrees Fahr. Inclusive. Analysis of Steel: Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33.

entrapment in the acicular ferrite and, consequently, reduced segregation of carbon in the austenite matrix. The final stages are, therefore, less inhibited than in the case of the 900 degrees Fahr. (480 degrees Cent.) reaction.

Hypereutectoid steel possesses similar transformation characteristics, with the exception that the transformation products are isothermally tempered in the latter stages of the reaction. This phe-

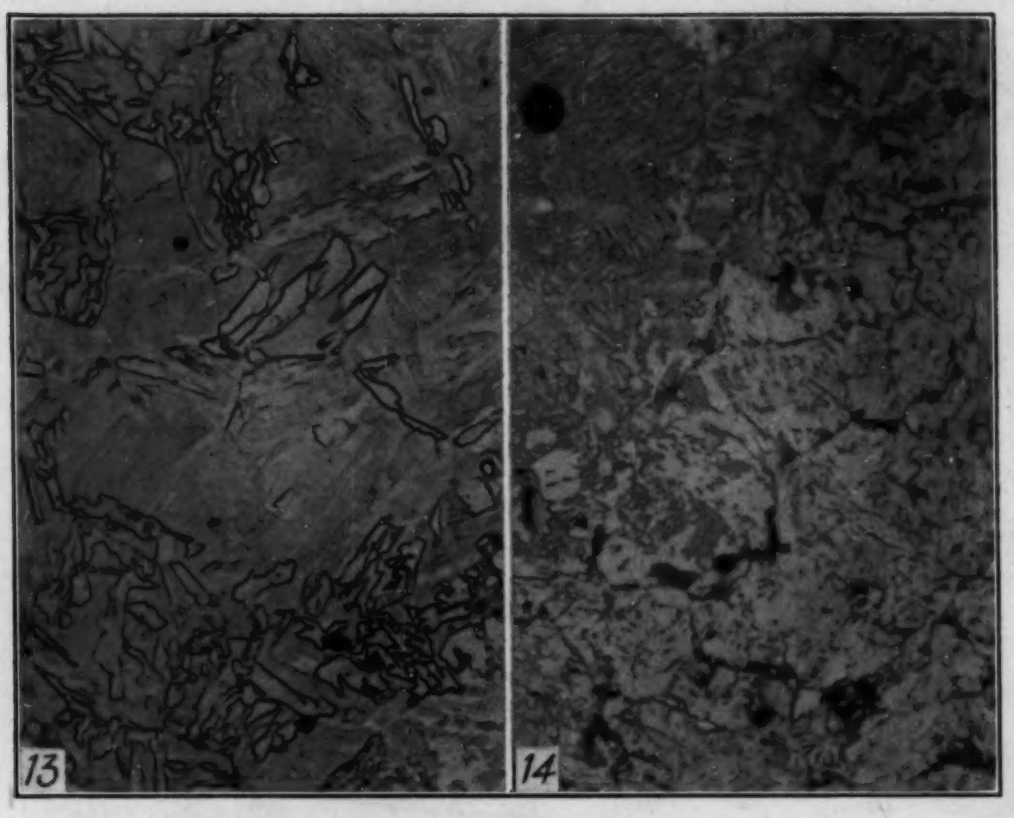


Fig. 13—Austenitized 1650 Degrees Fahr., 10 Minutes. Lead-Quenched to 900 Degrees Fahr., Held 17 Seconds, Water-Quenched. Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33. Approximately 25 Per Cent Transformed to Acicular Ferrite. Light Etching Background is Martensite Formed on Water Quench.

Fig. 14—Austenitized 1650 Degrees Fahr., 10 Minutes. Lead-Quenched to 900 Degrees Fahr., Held 72,000 Seconds, Alcohol-Quenched. Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33. Approximately 90 Per Cent Transformed to Ferrite Carbide Aggregate. Gray Structure is Martensite Formed on Water Quench. Both Specimens Etched in 1 Per Cent Nital, 10 Seconds. × 1000.

nomenon was not observed for the hypoeutectoid steels. The 500 degrees Fahr. (260 degrees Cent.) transformation, Fig. 10, reveals this behavior by a contraction after transformation is apparently complete.

3. Martensite Formation Followed by Acicular Transformation of Remaining Austenite—For dilation curves below 690 degrees

Fahr. (365 degrees Cent.) for the 0.30 per cent carbon alloy and below 320 degrees Fahr. (160 degrees Cent.) for 1 per cent carbon steel, the initial contraction differs from stage "a" of the curves discussed above; the length change from 1650 degrees Fahr. (900 degrees Cent.) to bath temperature decreases rather than increases with lowered temperature. This indicates that the specimen does not reach bath temperature untransformed, as for previous cases, but

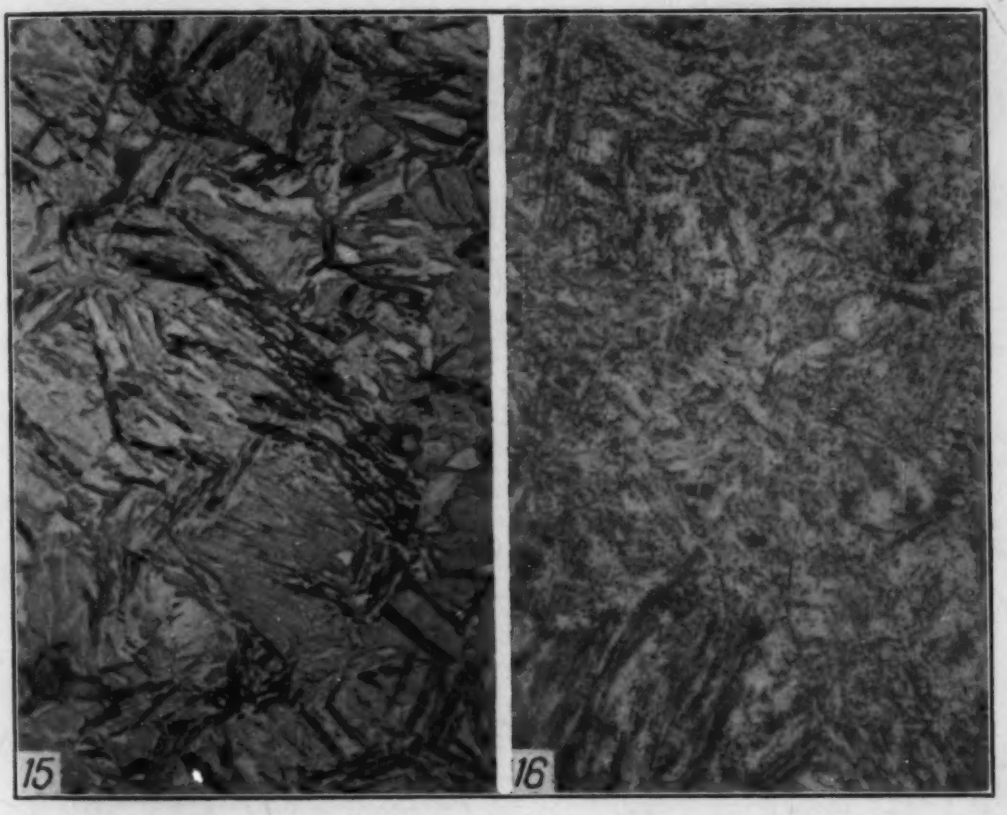


Fig. 15—Austenitized at 1650 Degrees Fahr., 10 Minutes. Lead-Quenched to 700 Degrees Fahr., Held 35 Seconds, Water-Quenched. Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33. Approximately 25 Per Cent Transformed to Acicular Structure. Remainder is Martensite Formed During Water Quench. Etched in 1 Per Cent Nital, 10 Seconds. × 1000.

Fig. 16—Austenitized at 1650 Degrees Fahr., 10 Minutes. Lead-Quenched to 700 Degrees Fahr., Held 6600 Seconds, Alcohol-Quenched. Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33. Completely Transformed to Acicular Structure. Etched in 2 Per Cent Nital, 7 Seconds. × 1000.

rather that a transformation involving expansion has accompanied the austenite contraction. This is the austenite-martensite reaction, which, as demonstrated by Greninger (2), is not suppressed even for very rapid cooling rates. The martensite point will be examined more fully in a later section (*"Summary Curves"*).

At the conclusion of austenite contraction + martensite formation (stage "a"), the dilation apparently follows the acicular reac-

tion pattern during the transformation of the remaining austenite. The curves of Fig. 10, 1 per cent carbon alloy steel at 300 degrees Fahr. (150 degrees Cent.), and of Fig. 12, 0.30 per cent carbon alloy steel at 600 and 500 degrees Fahr. (315 and 260 degrees Cent.), indicate the following steps:

a. Simultaneous austenite contraction + martensite formation.
b. A short incubation period of austenite remaining from (a),

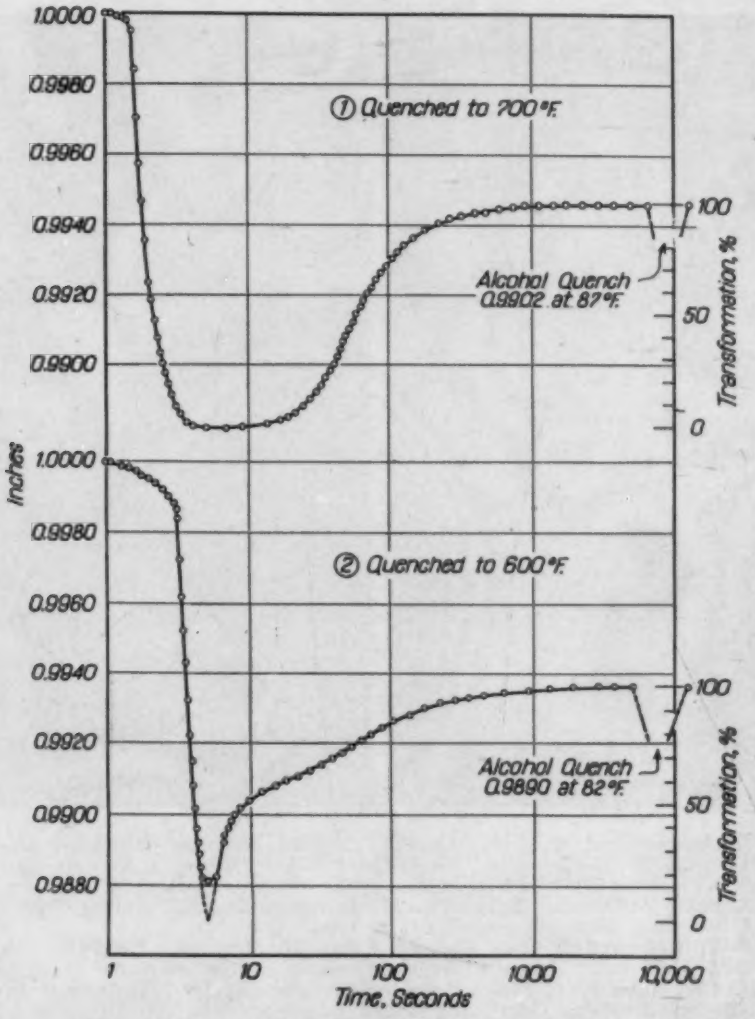


Fig. 17—Progress of Transformation as Interpreted from Dilatometer Curves. Analysis of Steel: Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33.

with perhaps minor amounts of transformation to acicular structure.

c. Rapid transformation to acicular structures.
d. Slow transformation to acicular structures.
e. Either period of no change or contraction by tempering.

As the temperature of the quenching bath is lowered, the amount of martensite formed increases and, as a corollary, the quantity of isothermal product diminishes. Furthermore, the temperature range for martensite formation varies with carbon content. For the 0.30 per cent carbon steel, 99 per cent of the austenite transforms to martensite on cooling to 300 degrees Fahr. (150 degrees Cent.) while, in the case of the 1 per cent carbon steel, the martensite reac-

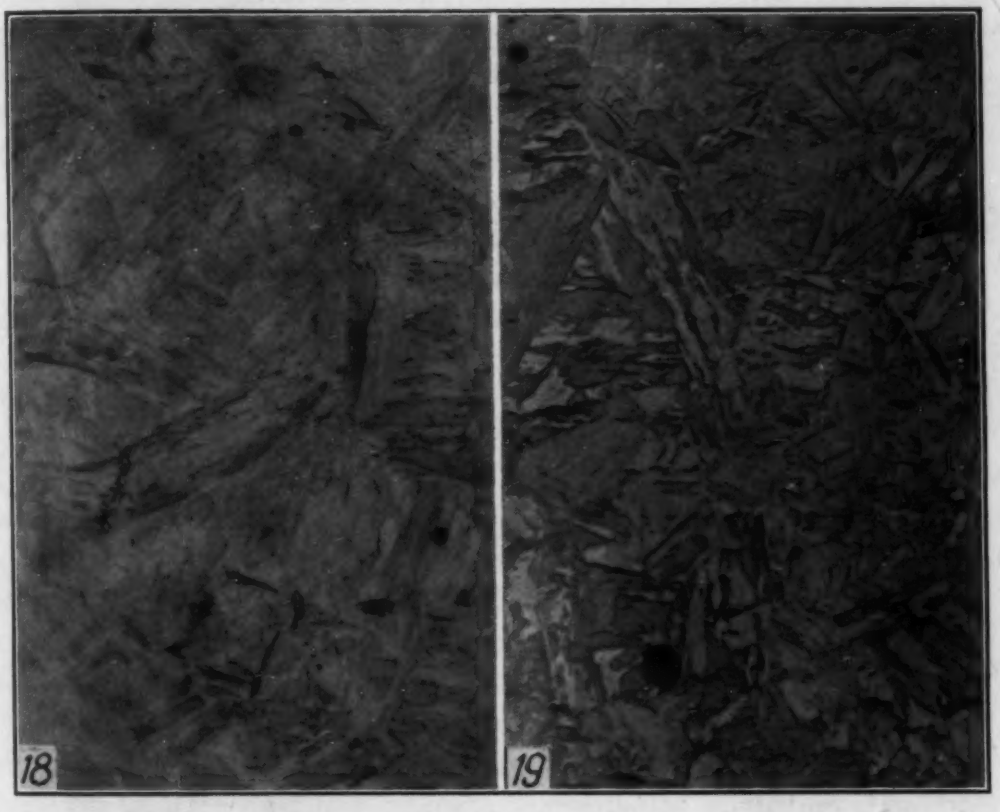


Fig. 18—Austenitized at 1650 Degrees Fahr., 10 Minutes. Lead-Quenched to 600 Degrees Fahr., Held 6 Seconds, Water-Quenched. Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33. Approximately 50 Per Cent Transformed to Martensite During Lead Quench. In the Succeeding Six Seconds a Trace of Acicular Product Appeared. Remainder Transformed to Martensite in Water Quench. Note That the Martensite Formed in the Two Different Temperature Ranges Cannot be Distinguished. Etched in 1 Per Cent Nital, 12 Seconds. × 1000.

Fig. 19—Austenitized at 1650 Degrees Fahr., 10 Minutes. Lead-Quenched to 300 Degrees Fahr., Held 3800 Seconds, Alcohol-Quenched. Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33. Completely (99 Per Cent) Transformed to Martensite While Cooling to Bath Temperature. Etched in 1 Per Cent Nital, 10 Seconds. × 1000.

tion is complete at some temperature below 70 degrees Fahr. (20 degrees Cent.) but above —120 degrees Fahr. (—84 degrees Cent.).

The dilation curves may be applied to ascertain the progression of martensite formation with temperature (Fig. 17). Since specimen length at 1650 degrees Fahr. (900 degrees Cent.) is known, the

length at bath temperature (if no transformation occurred on quenching) may be calculated. The difference between this mathematical value for austenite at bath temperature and the fully transformed length is the total transformation expansion. The portion attributed to martensite formation is, hypothetically, the expansion during cooling to bath temperature. Therefore, the difference between the theoretical total expansion and that actually recorded by the dilatometer may be regarded as a measure of the martensite formed during each quench.

The point of ending of martensite formation and beginning of the acicular reaction is, however, not always distinct. For example, in Fig. 10, 1 per cent carbon steel at 300 degrees Fahr. (150 degrees Cent.), marked acicular transformation ensues only well after the completion of martensite production while, in Fig. 12, 0.30 per cent carbon steel at 600 degrees Fahr. (315 degrees Cent.), the martensite apparently nucleates the acicular reaction. For the purposes of this research, all expansion for the first 5 seconds, after passing 1350 degrees Fahr. (730 degrees Cent.), has been considered as martensite formation for quenches below the "M" point. This procedure insures that the specimen is at bath temperature and that all martensite formation is included. Only a small error can be introduced by any isothermal transformation in this period. These methods are employed for investigating the progression of martensite formation; the beginning point is determined by practices discussed later.

The photomicrographs indicate some metallographic difference between the acicular and martensitic types of transformation. Although the partly transformed acicular structure of 0.30 per cent carbon alloy steel, Fig. 15, bears a resemblance to the martensitic structures of Figs. 18 and 19, the fully transformed acicular structure is quite different, Fig. 16. The reason for this apparent anomaly is that the acicular structure is formed by a process of nucleation and growth; the needles, which were originally distinct against a martensitic background of different etching characteristics, have grown and coalesced to provide the structure of Fig. 16. In contrast, the martensitic structure, resulting from a shear mechanism (2), (13), retains its needle-like appearance.

Summary Curves

The dilation curves provide basic data for the quantitative de-

scription of structural changes which transpire during cooling from the austenite field. This approach may be summarized in the following series of curves:

1. Austenite Contraction and the "M" Point.
2. "M" Point Versus Per Cent Carbon.
3. The "S"-Curve.

1. Austenite Contraction and the "M" Point—The values of the austenite contraction to bath temperature, stage "a", when plotted against temperature, Fig. 20, describe an approximately straight line until the "M" point temperature is attained. This represents a coefficient of contraction of 12.2×10^{-6} in./in./°F. for austenite. When contractions to lower temperatures are plotted, a second line at approximately 90 degrees to the first is obtained; their intersection establishes the "M" point. The actual values of the minima for quenching to temperatures below the "M" point have no fundamental significance; they are merely the points at which the contraction of cooling austenite has been counteracted by the expansion of transforming austenite.

The data probably constitute a straight line below the "M" point because of uniform reproducibility of the quenching procedure. The intersection, however, delineates fairly the significant martensite point as the temperature at which maximum austenite contraction is obtained (stage "a").

It is at first surprising that the amount of maximum austenite contraction should apparently decrease below the "M" point rather than remain constant. It would be anticipated that the specimen should first contract to a length characteristic of the "M" point temperature and then expand. An explanation may appear in the longitudinal temperature gradients which exist in the specimen during quenching. For example, since immersion from end to end requires about 0.4 second and since the rate of cooling of infinitesimal individual sections of the coupon is 7000 degrees Fahr. (3890 degrees Cent.) per second, a considerable longitudinal gradient (500 to 600 degrees Fahr.) exists. The lower portions of the specimen first immersed in the bath transform to martensite while the upper portions remain above this critical temperature. Because martensite, even at low temperature, is more voluminous than austenite at "M" point temperature, full contraction is never obtained with this procedure. For example, from Curve 2, Fig. 12, the 1-inch dilatometer specimen has contracted as austenite to 0.9880 inch at 700 degrees

Fahr. (370 degrees Cent.) while the length of a more voluminous martensitic specimen at 86 degrees Fahr. (30 degrees Cent.) is 0.9897 inch (Curve 5).

The longitudinal gradient was established as the more important of these effects by providing for an instantaneous quench as follows: A vertically split iron tube closed at the bottom, of sufficient bore to accommodate the lower portion of the dilatometer, was positioned

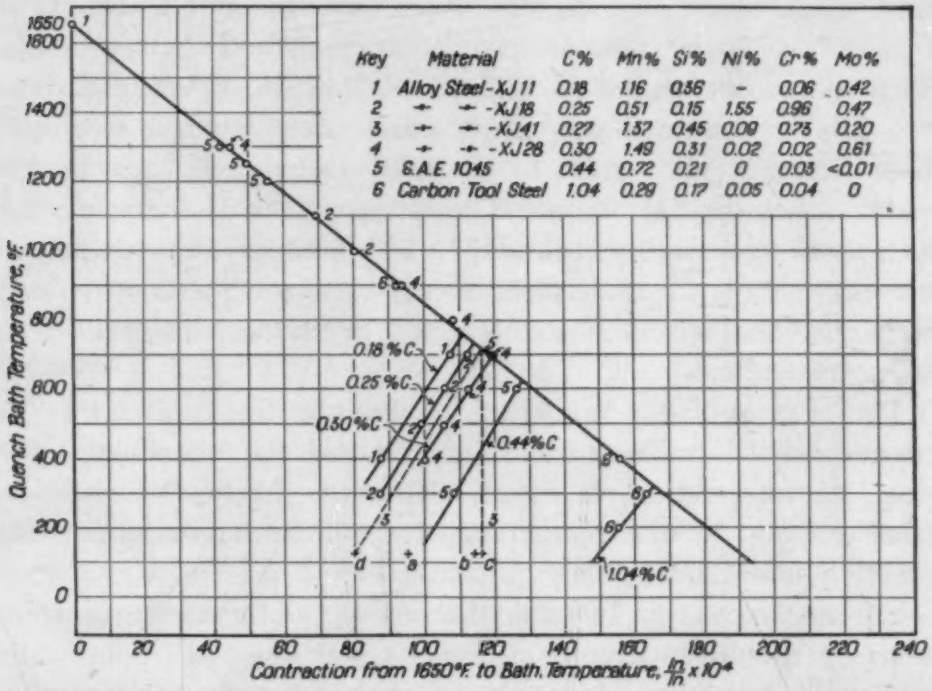


Fig. 20—Graphical Determination of "M" Points by Means of Contraction Observed During Quench.

in the quenching bath to provide a sub-surface air chamber. After austenitizing the dilatometer specimen (0.27 per cent carbon steel, see Fig. 3), the pot containing this chamber was swung into place and the tube sprung open. By this technique the quenching medium made contact with the entire length of the specimen at practically the same instant. A longitudinally uniform quench was thus obtained.

In the first experiments with this method the quenching medium was Wood's metal at 200 degrees Fahr. (95 degrees Cent.). The shock to the quartz, however, by the sudden rush of swirling metal upon opening the tube, was too great and the dilatometer was fractured. To avoid this difficulty an oil bath at 122 degrees Fahr. (50 degrees Cent.) was successfully used, yielding the point marked

(3-b) of Fig. 20. The expected contraction, calculated for the "M" point length (point "c", Fig. 20), was closely approached by the actual value (point "b"). Point "a" represents the contraction for the usual quench wherein a longitudinal gradient exists. Since both these experiments were conducted at 122 degrees Fahr. (50 degrees Cent.), which is below the temperature for complete martensite formation for this steel, point "a" represents a greater value than

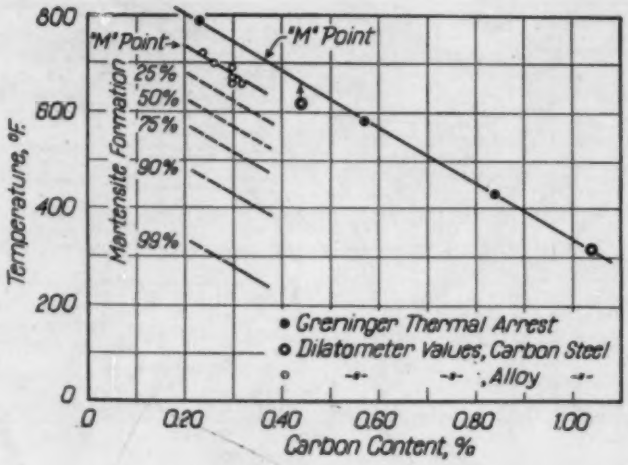


Fig. 21—Variation of Martensite Formation Characteristics With Per Cent Carbon Content.

indicated by extrapolating for 0.27 per cent carbon (point "d"). This is the result of martensite contraction after transformation is completed on cooling.

2. "M" Point Versus Per Cent Carbon—In Fig. 21 the temperatures of various "M" points have been plotted against carbon content. The tool steel (1.04 per cent carbon) conforms closely to Greninger's data for the same type steel. The value for S.A.E. 1045 displays a certain divergence. The alloy steels (see Fig. 20 for chemical analyses) have been considered in a separate category. Since the carbon range is not sufficient to delineate a well-defined slope, the line has been drawn parallel to Greninger's relationship.

In addition to the "M" point, the dilatometric method also permits estimation of the temperatures for various degrees of martensite formation during the quench as already discussed. The data have not yet been completely verified but tentative values have been indicated in the dotted lines of Fig. 21.

3. The "S"-Curve—Fig. 22 illustrates a typical S-curve for a medium carbon alloy steel which has been derived from dilatometric

data presented earlier in the report. For the ferrite-pearlite range, the curves represent the per cent of total isothermal length change rather than the per cent of either particular transformation product. Both phases could have been treated separately by proper analysis of the dilatometer curves but it was considered that the graph then became too complicated.

Derivation of the dotted lines representing per cent martensite

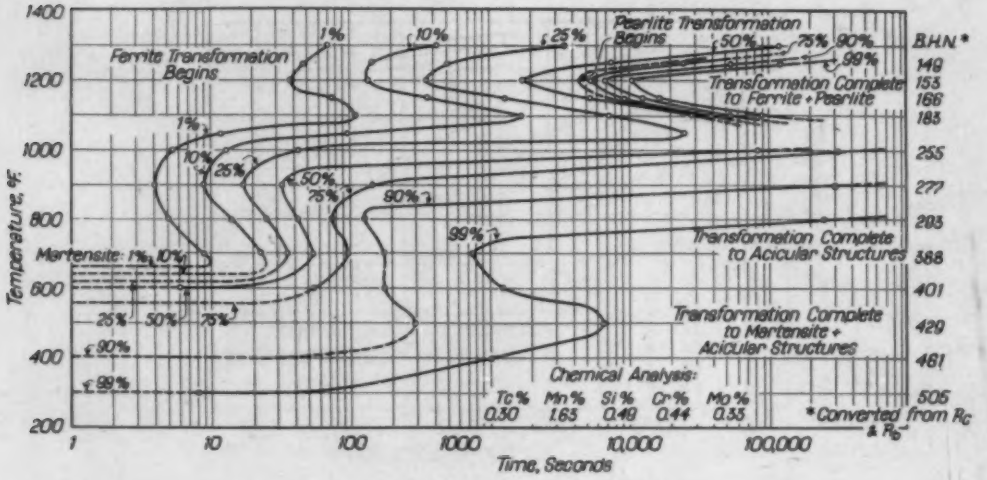


Fig. 22—Transformation Characteristics of an Alloy Steel. Analysis: Carbon 0.30, Manganese 1.63, Silicon 0.49, Chromium 0.44, Molybdenum 0.33.

transformation has been discussed. They are intended to represent transition from an acicular isothermal reaction rate to the rapid martensite transformation.

Since the use of the S-curve in heat treatment has received thorough attention elsewhere, only a few significant features of Fig. 22 will be considered.

The upper nose, with a comparatively rapid reaction rate, indicates the possibility of a time-saving annealing cycle, consisting of austenitizing, moderately rapid cooling to 1200 degrees Fahr. (650 degrees Cent.), holding at this temperature for 3 hours, and rapid cooling. A tediously long continuous cool from the austenite field would otherwise be required to accomplish the same degree of softening. This type of treatment was discussed as early as 1922 by Whiteley (14), by Dopfer and Wiester (15) in 1935, by Kontorovich (16) and Gudzon and Sumin (17) in 1936 and Payson (18) in 1939. It is mentioned again because the sharpness of the upper nose indicates that the annealing temperature must be carefully selected and controlled for softening alloys of this type.

The careful delineation of the martensitic range provides data for avoiding the rapid and voluminous martensite transformation wherever minimized quenching stresses rather than maximum hardness are required.

Conclusions

The advantages of this dilatometer exist in the more precise seating of the specimen inherent with a tension mount, in the velocity of quenching obtained by the avoidance of shrouding the sample by the supporting parts, but principally in the provision for a complete record of length changes during the entire heat treatment. The equipment may also be applied for recording expansion and contraction curves in the determination of equilibrium critical points. It is suggested that the device offers an interesting approach to studies of the relation of chemical analysis to volumetric behavior during austenite transformation. A novel method for the rapid and precise delineation of the martensite point has been proposed.

The comments and helpful suggestions of Professors Morris Cohen and Victor O. Homerberg of the Massachusetts Institute of Technology are gratefully acknowledged.

Bibliography

1. E. S. Davenport and E. C. Bain, "Transformation of Austenite at Constant Sub-Critical Temperatures," *Transactions,* American Institute of Mining and Metallurgical Engineers, Iron and Steel Division, Vol. 90, 1930, p. 117.
2. A. B. Greninger and A. R. Troiano, "Kinetics of the Austenite to Martensite Transformation," Transactions, American Society for Metals, Vol. 28, No. 3, 1940.
3. D. W. Murphy, W. P. Wood and D. Girardi, "The Rate of Austenite Transformation in Cast Iron," Transactions, American Society for Metals, Vol. 26, 1938, p. 173.
4. R. M. Parke and A. J. Herzig, "Hardenability of Molybdenum S.A.E. Steels," *Metals and Alloys,* Vol. 11, 1940, p. 6.
5. E. C. Bain, "The Process and Result of Austenite Transformation at Constant Temperature," *Metals and Alloys,* Vol. 8, 1937, p. 22.
6. R. A. Flinn, Morris Cohen and John Chipman, "The Acicular Structure in Nickel-Molybdenum Cast Irons," Transactions, American Society for Metals, Vol. 30, 1942, p. 1255.
7. E. S. Davenport, "Isothermal Transformation in Steel," Transactions, American Society for Metals, Vol. 27, 1939, p. 837.
8. W. T. Griffiths, L. B. Pfeil and N. B. Allen, "Intermediate Transformation in Alloy Steels," British Iron and Steel Institute, Special Report No. 24, p. 369.
9. R. F. Mehl, "The Physics of Hardenability," American Society for Metals Symposium on Hardenability of Alloy Steels, 1938, p. 1.
10. "Tentative Method of Test for Linear Expansion of Metals," *Proceedings,* American Society for Testing Materials, Vol. 34, Part I, 1934, p. 33.

11. P. Chevenard, "Mecanisme de la trempe des aciers au carbone" (Mechanism of the Carbon Steels), *Revue de Metallurgie, Mem,* Vol. 16, 1919, p. 17-79.
12. Donald B. Oakley and Joseph F. Oesterle, "Dilatometric Studies in the Transformation of Austenite in a Molybdenum Cast Iron," TRANSACTIONS, American Society for Metals, Vol. 28, 1940, p. 832-852.
13. S. Epstein, Alloys of Iron and Carbon, Vol. I Constitution, p. 212.
14. J. H. Whiteley, "The Formation of Globular Pearlite," *Journal,* Iron and Steel Institute, Vol. CV, 1922, p. 340-357.
15. Dopfer and Wiester, "Influence of Alloying Elements on the Transformation of Austenite and the Tensile Properties of Alloy Steel Heat Treated in Steps," *Archiv Eisenhüttenwesen,* June 1935, p. 541-546.
16. I. E. Kontorovich, "Isothermal Transformation of Austenite in Alloy Steels and Mechanical Properties Produced Thereby," *Kachestvennaya Stal,* Vol. 4, No. 8, 9, 1936, p. 13-18.
17. Prof. N. T. Gudzov and I. A. Sumin, "Step Hardening of Die Steel," *Metallurgy,* Vol. 2, No. 8, 1936, p. 40-52.
18. Payson, U. S. Patent Nos. 2,188,155 and 2,251,289.

DISCUSSION

Written Discussion: By Morris Cohen, associate professor of physical metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

Those of us who have had some experience in the application of dilation measurements to phase-transformation studies can readily appreciate the ingenuity of these investigators in their development of a reliable high-speed quenching dilatometer. Such an instrument is not only of immense value in research on the kinetics of austenite decomposition but can be used equally effectively in tempering and precipitation studies where "instantaneous" heating to the reaction temperature is desirable. Rates of austenitizing can also be followed closely in this dilatometer, provided the creep factor does not unduly complicate the length changes.

The present writer is particularly impressed with the authors' methods for deriving quantitative information from their dilatometric data, and is gratified to note that the martensite range shown in the S-curves of Fig. 22 is identical in principle with that first proposed by the writer elsewhere.[1, 2] High speed steel also exhibits this type of behavior,[3] as well as the double-nose isothermal transformation curves.

With regard to the nature of acicular ferrite, Mr. Sidney Siegel has recently demonstrated[4] that an appreciable build-up of carbon in the remaining austenite results from the formation of such ferrite. This work was conducted on a 0.6 per cent carbon-5 per cent nickel steel, using lattice parameter measurements on the retained austenite and the tetragonal martensite present in the steel

[1]M. Cohen, Discussion of paper by A. B. Greninger and A. R. Troiano, TRANSACTIONS, American Society for Metals, Vol. 28, 1940, p. 563.

[2]M. Cohen, Discussion of paper by E. R. Saunders and J. F. Kahles, TRANSACTIONS, American Society for Metals, Vol. 30, 1942, p. 1155.

[3]P. Gordon, M. Cohen, and R. S. Rose, "The Kinetics of Austenite Decomposition in High Speed Steel," published in this issue of TRANSACTIONS, page 161.

[4]S. Siegel, "An X-ray Study of Acicular Ferrite in Five Per Cent Nickel Steel," Massachusetts Institute of Technology Bachelor's Thesis, Department of Metallurgy, Jan. 1943.

after partial isothermal transformation and quenching to room temperature. Also in line with the authors' ideas is the fact that Mr. Siegel found no carbon build-up in the austenite when the hot quenching temperature was sufficiently low to result in dark etching acicular bainite.

It is hoped that the authors will find it possible to continue their dilatometric studies on the fundamentals of austenite decomposition. Despite the voluminous literature on this important subject, much remains to be done.

Written Discussion: By Edgar C. Bain, assistant to vice-president, United States Steel Corp., Pittsburgh.

Messrs. Flinn, Cook and Fellows have reported in a most clearly written style their experiences with the determination of transformation time-patterns for austenite and their design of another version of the quenching dilatometer. That they have become exceedingly observant and therefore thoroughly familiar with this technique is assuringly evident from the paper.

It would appear that they have built a good dilatometer and the results indicate that they have justified their somewhat unexpected preference for moving many pounds of quenching material to moving a light dilatometer with specimen. This choice of operation would seem to be a matter of strictly personal preference.

It should be pointed out that a dilatometer, made of steel, and positively positioned in the quenching bath for a sufficient time to induce the steady state of heat flow is also satisfactory but, in this case, the specimen must be independently heated and quickly introduced. This operation must be accomplished in a time interval short enough to avoid any transformation of austenite in the specimen prior to the quench and sufficiently brief also to avoid significant upset of the steady state in the dilatometer. To fulfill these requirements great dexterity is needed and the original work at the United States Steel Corporation Research Laboratory shows that adequate proficiency is soon acquired. Nevertheless, quick insertion of the heated specimen more or less necessitates a specimen brought into compression by the dilatometer feeler; tension is generally better if the specimen is well designed, since in this case it may be much thinner. If thinner, then steels of lower hardenability can be properly quenched in a lead bath.

Several fused-quartz dilatometers were built by us and used in the manner of the present paper except that the dilatometer (plus specimen) was moved from the austenitizing bath to the quenching and transformation bath; this seems faster to us, but may not necessarily be so for others. Some attack by the bath usually embrittled the silica. In any event, the positioning for photographing the clock and dial is equally efficacious by either technique since the dilatometer is easily brought against stops. The simultaneous photography of clock and dial was entirely successful in a comparatively crude set-up reported by Waring and Bain in 1928 (TRANSACTIONS, American Society for Steel Treating, Vol. 15, p. 69).

With the concurrent availability of their improved dilatometer and the clear intent of the authors to make a strictly quantitative deduction from dilatometer indications it may seem desirable to them to turn to a clarification of the relation between the length change of long, thin specimens and the volume change during transformation. Quite clearly in the general case the length of any one

dimension does not always faithfully follow the cube root of the volume (or inversely the density) during change. Nor indeed is it to be expected that the length of a dilatometer specimen of tempered martensite will always be precisely that from an originally identical specimen which transforms to fine pearlite or bainite. These details certainly do not constitute any valid objections to the plotting of "S-curves" from dilatometric data in the ordinary way, but are marginal fields which may cast some light upon such interesting items as the persistent and systematic difference between the time-transformation curves for bainite (around 600 degrees Fahr.) as determined respectively by the dilatometer and by microscopic examination. This difference is small but definite and completely reproducible and always in the same direction.

Perhaps we shall one day find that, rapid though the transformation to martensite is, it is not really instantaneous and that a small measurable time elapses in the formation of the particular proportion of this constituent, according to temperature, before this reaction ceases or the more tardy bainite reaction sets in.

It is a very welcome circumstance to find additional investigators with the mature viewpoint of the present authors extending the list of steels for which the so-called S-curves are to be available.

Written Discussion: By R. M. Parke, chief research metallurgist, Climax Molybdenum Co., Detroit.

The authors of this paper have presented data on the transformation of austenite which have immediate practical application. The insensitivity of the coefficient of expansion of austenite to large variations in temperature, comparatively large variations in carbon content, and moderate variations in alloy content seems surprising. However, this conclusion is essentially confirmed by data possessed by our laboratory.

It would be of interest to learn whether the authors measured the length change per unit length caused by complete transformation of austenite at constant subcritical temperatures, and whether they attempted to relate such data to the temperature of transformation.

Written Discussion: By Otto Zmeskal, research metallurgist, Universal-Cyclops Steel Corp., Bridgeville, Pa.

The authors are to be congratulated on this most interesting and illuminating paper. The design of their dilatometer which permits a complete record of length changes in a specimen during an entire heat treating cycle will be welcomed by the investigators in this field.

The authors used 1650 degrees Fahr. (900 degrees Cent.) as their austenitizing temperature. Have they observed the effect on their apparatus of heating to higher temperatures as 1800 to 2200 degrees Fahr. (980 to 1205 degrees Cent.)? Would not creep greatly affect the results obtained by the use of this dilatometer at these temperatures? Would the quartz parts stand the shock of quenching from these temperatures into the low temperature transformation baths? Would not an insertion of the thin specimen in the transformation bath at an angle only slightly off from vertical result in spurious readings of the dilatometer? The buoyancy effect of the displaced metal conceivably could bend the specimen. Might not the discontinuities in the (a) segments

of curves 2, 3, 4 and 5 of Fig. 10 and curves 1, 2, 3, 4 and 5 of Fig. 12 be due to this action?

A constant austenitizing time of 10 minutes was used in the experiments reported. Was any study made of the effect of longer austenitizing times, i.e., of the effect of the homogeneity of the austenite on transformation?

Authors' Reply

We wish to thank Dr. Cohen for his generous discussion. We have used the dilatometer for "instantaneous" heating to temperature for tempering studies as suggested and obtained satisfactory results. Also, as mentioned by Dr. Cohen, austenitizing rate may be recorded without any change in the apparatus. The additional data regarding carbon distribution during acicular bainite formation are most illuminating.

Discussion by Dr. Bain provides an almost indispensable addition to any report of austenite transformation and is especially welcome to us. We agree that choice of dilatometer apparatus is a matter of personal preference and the reasons for our selection have been covered by the paper and by Dr. Cohen's discussion. For the sake of investigators who may consider using a dilatometer of the type described here, it should be added that the life of the inner silica tube under the conditions described was of the order of fifty runs and the outer tube did not require replacement.

The difference between the data from dilatometric and microscopic determination of time-transformation curves around 600 degrees Fahr. (315 degrees Cent.) may be explained if the discrepancy referred to by Dr. Bain occurs in steels of relatively low carbon content, i.e., approximately 30 per cent carbon. If 600 degrees Fahr. (315 degrees Cent.) is below the "M" point, the dilatometer will provide a transformation curve for the bainite reaction alone, since the martensite reaction will be complete by the time the specimen reaches isothermal transformation temperature. In the case of the microspecimen method, however, specimens subsequently quenched to room temperature before initiation of the bainite reaction may be expected to show evidences of transformation if the martensite formed on cooling to 600 degrees Fahr. (315 degrees Cent.) is tempered at temperature. As explained in the paper, *percentage* transformation to martensite in this case may be clearly determined by the dilatometric method from the diminished austenite contraction on quenching to 600 degrees Fahr. (315 degrees Cent.).

Dr. Parke's confirmation of the insensitivity of the coefficient of expansion of austenite to variations in temperature range and chemistry is appreciated.

The length change accompanying austenite transformation at subcritical temperatures has, of course, been observed for each specimen quenched as illustrated by the typical dilatometer curves presented in the paper. It may be stated in general that, above the "M" point, the expansion tends to vary inversely with isothermal transformation temperature. There is also some suggestion of a similar inverse trend of expansion with change in carbon content. The following table lists examples of these behaviors for the types of steel discussed.

Isothermal Transformation Expansion—Inches per Inch

Alloy Steel

Carbon Per Cent	1200 Degrees Fahr.	1000 Degrees Fahr.	800 Degrees Fahr.
0.30	39×10^{-4}	48×10^{-4}	56×10^{-4}

Carbon Steels

Carbon Per Cent	1300 Degrees Fahr.	1200 Degrees Fahr.	900 Degrees Fahr.	700 Degrees Fahr.
0.45	34×10^{-4}	33×10^{-4}	42×10^{-4}	45×10^{-4}
1.04	10×10^{-4}	11×10^{-4}	17×10^{-4}	33×10^{-4}

We have not as yet investigated a sufficient number of steels differing solely in carbon content to attempt a graphical interpretation of this phenomenon. The data also intimate a marked difference in behavior between distinct alloy classifications.

Dr. Zmeskal's comments and interest are acknowledged. Quenching temperatures in the 1800 to 2200 degrees Fahr. (980 to 1205 degrees Cent.) range have been avoided because of the question of grain growth. Protection of the molten metal bath from oxidation becomes a problem at these temperatures. The matter of creep is not particularly serious since the amount of flow occurring during the transfer to the lower temperature bath and in the actual quench will be infinitesimal in nearly every case. Using the recorded length at the instant of transfer should thus provide an acceptable reference value for determining austenite contraction. Creep at high austenitizing temperatures would be troublesome only if the elongation occurring during the soak later caused the register of isothermal expansion to extend beyond the span of the dial gage scale.

The quartz parts have successfully withstood quenches from 1650 to 70 degrees Fahr. (900 to 20 degrees Cent.) in water. No data are available beyond this range.

The discontinuities at points (a) in Figs. 10 and 12 are merely a break in the scale used in plotting to permit reproduction of the essential portions of the dilatometer curves within a reasonably sized graph. Inspection of the units along the ordinate should make this clear. The complete curves exhibit an entirely smooth and continuous line in this location. There has been no evidence of bending.

BURSTING TESTS ON NOTCHED ALLOY STEEL TUBING

By G. Sachs and J. D. Lubahn

Abstract

Heat treated alloy steel tubing subjected simultaneously to circumferential and longitudinal tension showed slightly higher tensile strengths and much lower ductility values in the circumferential direction than rod tested in tension.

A sharp longitudinal notch on the outside surface of the tubing was found to be the most severe embrittling agent observed so far, in that steels with a strength level as low as 150,000 pounds per square inch had a notch strength less than the strength of unnotched tubing.

Such a single notch did not have the strength-increasing effect which was expected for sufficiently ductile conditions. A double notch (on both inside and outside surface), however, did produce such a strength-increasing effect, but the increased lateral stress which is responsible for such an effect did not cause any greater embrittlement than a single notch.

The conditions of fiber direction are usually considered to be less favorable in tubular specimens machined from solid rod and subjected to internal pressure than for drawn tubing. However, no such effect of fiber direction was observed in tests on notched tubes machined from rod.

Test Procedure

A POSSIBILITY of creating a multiaxial stress state is offered by tests on thin-walled tubular specimens which are simultaneously subjected to internal pressure, which creates circumferential tension, and to longitudinal tension. The ratio of the two tensions can be varied almost anywhere between 1 to 0 and 0 to 1. The third principal stress is a small radial compression, on the average, of one-half of the internal pressure, and it is insignificant. Such tests have

This paper is a report on part of the research program conducted at Case School of Applied Science under the sponsorship of the International Nickel Company, Inc.

A paper presented before the Twenty-fourth Annual Convention of the Society held in Cleveland, October 12 to 16, 1942. Of the authors, G. Sachs is professor of physical metallurgy, and J. D. Lubahn is research assistant, department of metallurgical engineering, Case School of Applied Science, Cleveland. Manuscript received July 15, 1942.

been carried out for the particular purpose of revealing the effect of the stress state on the plastic flow (1), (2).[1]

It was decided to make some tests on tubing, selecting a stress state which should yield the minimum ductility. However, the maximum triaxiality present in any combined stress test on cylindrical tubing is probably rather small compared with that present in notched

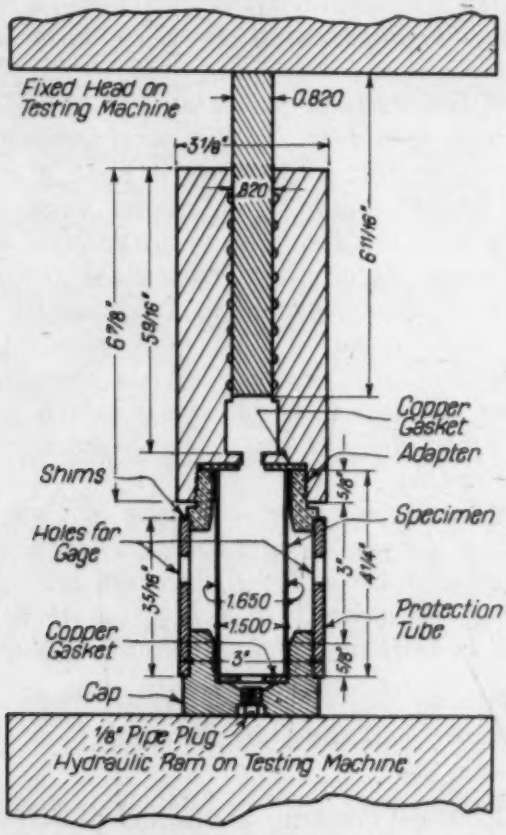


Fig. 1—Equipment for Testing Tubing Under Combined Internal Pressure and Longitudinal Tension.

tensile test bars, for instance. On the other hand, it should be possible to reach a particularly high triaxiality by the superposition of a notch effect on the triaxiality present in the tubing. Therefore, a number of additional tests have been carried out on tubing provided with a sharp longitudinal notch.

Difficulties were encountered in securing high pressure equipment which would resist the required maximum pressure of 30,000 pounds per square inch. The solution of the problem which was

[1]The figures appearing in parentheses refer to the bibliography appended to this paper.

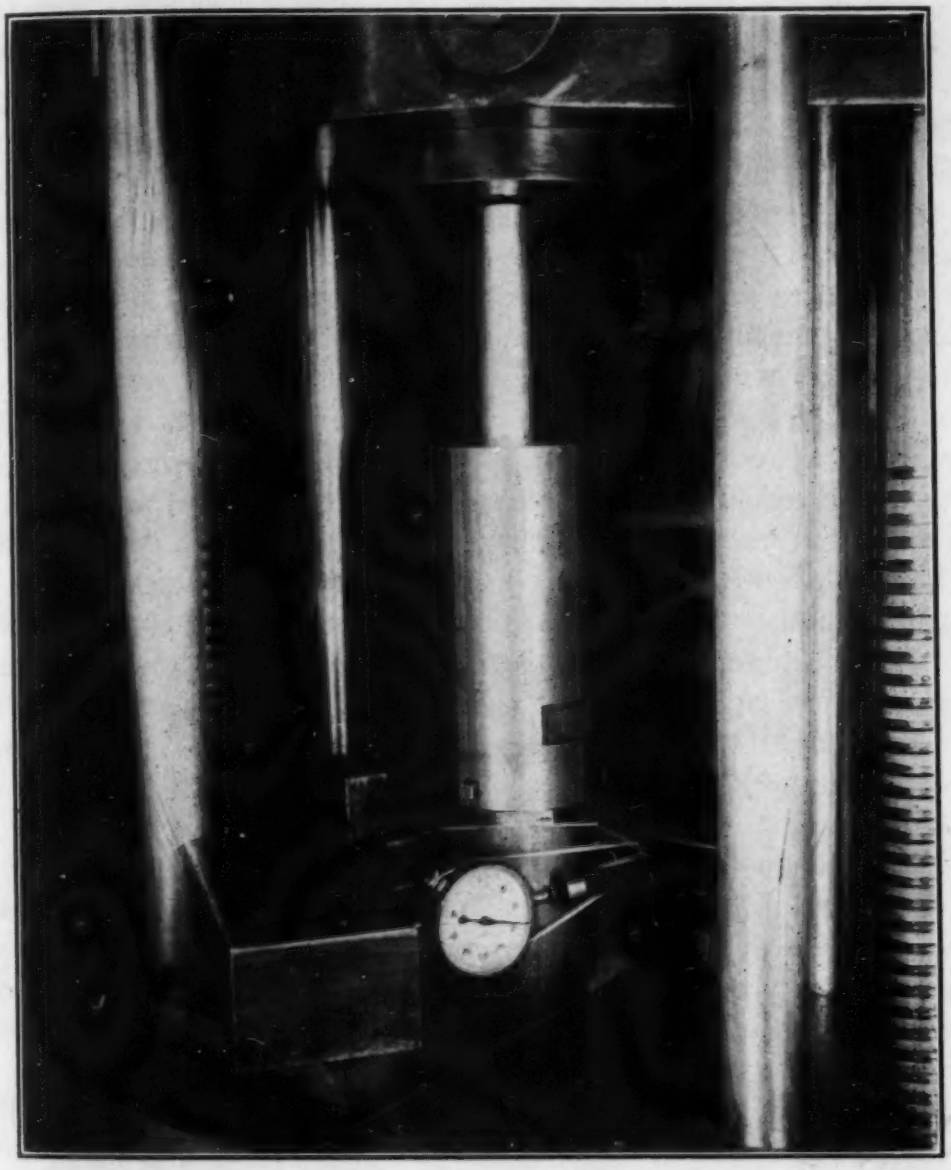


Fig. 2—Equipment for Testing Tubing Under Combined Internal Pressure and Longitudinal Tension.

finally adopted* is illustrated in Figs. 1 and 2. The high pressure generating chamber is a cylinder made from an air-cooled alloy steel, into which a hardened, high speed steel piston could be pushed. Both the cylinder and the piston were lapped, giving a few ten-thousandths clearance. One end of the high pressure cylinder contained a small opening, leading into a larger chamber. This chamber was provided with a threaded, heat treated steel adaptor, forming the one grip for

*We are indebted to Messrs. R. H. Smith and C. L. Harvey of Lamson and Sessions Company, Cleveland, Ohio, for their support in securing this equipment.

the tubular specimen. The other end of the specimen was closed by a threaded cap, having a recess in the bottom and a tap screw which served as an air valve in filling the assembly with a heavy cylinder oil. The assembly was placed into a 60,000-pound Olsen hydraulic testing machine serving both as pressure source and pressure gage.

A piece of heavy tubing surrounded the specimen, serving simultaneously as a protection device and as a spacer. The desired stress state is characterized by the feature that the length of the specimen does not change during the test. Instead of separately adjusting the longitudinal tension, therefore, the protection tube was fitted by means of spacing shims in such a manner that only a few thousandths longitudinal shortening were possible, which were used up in the elastic region. For this purpose, the specimen was set up in the apparatus in such a way that it was free to shorten by a small amount. This was done by using copper gaskets which were thick enough so that when the specimen was tight against them, there was some clearance between the protection tube and the corresponding shoulders on the adaptor and cap. Then sheet metal shims having a total thickness of 0.000 to 0.003 inch less than the above clearance were inserted into the space, thus reducing the clearance to less than 0.003 inch.

The diameter change during the test was measured with a specially built, lever-type gage, Figs. 2 and 3. It was found necessary to equip the gage with a safety wire, which permitted one anvil to be suddenly pushed through the arm of the gage in order to protect the Ames dial and the lever from damage when the specimen broke.

This anvil attained a considerable velocity upon fracture of the specimen, and a steel block was therefore set up in its path to prevent it from doing any harm.

The shape of the unnotched specimen is shown in Fig. 1. The notched specimen had a 45-degree longitudinal notch reaching over the total cylindrical length and having a depth between 30 and 40 per cent of thickness. Pieces of the tubing slightly over 4¼ inches in length were cut from the tubing supplied and roughly machined from $1\frac{23}{32}$ = 1.72-inch outside diameter, and ⅛ inch = 0.13-inch wall thickness to nearly the finished dimensions, allowing for grinding of the cylindrical surfaces and to a length of 4.250 ± 0.001 inches. After heat treatment, the specimens were ground on the outside to 1.650 ± 0.001 inch diameter, and honed on the inside to 1.500 ±

0.001 inch diameter using a collapsible hone, thus giving a wall thickness of 0.075 inch. The notch was also machined before heat treating, but finished after heat treating by scraping with a tool kept as sharp as possible by repeated grinding. For hardening, the tubes were packed in a mixture of charcoal and magnesia in a container

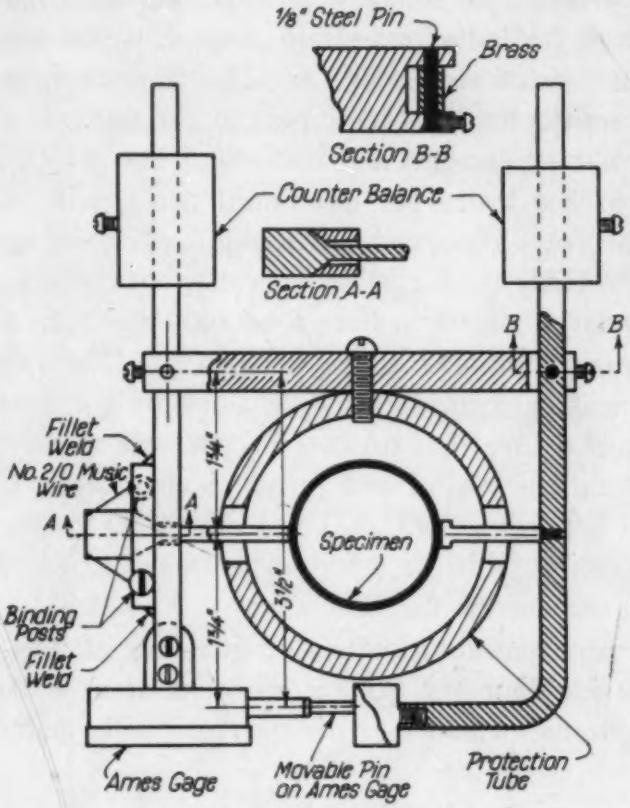


Fig. 3—Gage for Measuring Diameter Change in Testing Tubing Under Internal Pressure.

about 2½ inches deep, heated 3 hours in a Hevi-Duty furnace, quenched in an air lift oil tank, and tempered for ½ hour in a Lindberg air convection furnace.

Tests were made primarily on S.A.E. 2340 tubing, heat treated at a temperature of 1450 degrees Fahr. (790 degrees Cent.).

Bursting Tests on Unnotched Tubing

Before the test, the outside diameter of the tube was measured, and the wall thickness was determined with ball-micrometers. In all cases the value taken was the average of ten measurements. The tubes were usually out of round by 0.002 to 0.003 inch and occa-

sionally by as much as 0.006 to 0.007 inch. Variations in wall thickness amounted to a maximum of 0.003 inch, or ± 3 per cent of the average wall thickness.

During the test, readings of change in diameter were taken for load increments of 500 pounds until yielding began, and then load readings were taken for diameter change increments of 0.0025 inch. These readings yielded stress-strain curves of the common appearance for heat treated steel, Fig. 4. The first straight line is fairly steep, and it represents the elastic part of the test. It changes slowly into another almost straight and horizontal line which represents the plastic part of the test. No maximum point, with subsequent decrease in load, was observed as in regular tensile tests of ductile materials. Probably such a point did exist, but could not be observed because the energy stored in the oil brought the tube suddenly from the point of maximum load to that of fracture. The value of modulus of elasticity was calculated from the slope of the first straight line in order to make sure that no loss of pressure occurred because of friction between the piston and pressure chamber. The values obtained varied between 28,000,000 and 32,000,000 pounds per square inch, thus agreeing with the common value of 30,000,000 within the limits of accuracy of the method.

The circumferential ultimate strength (U) of thin-walled tubing having an outside diameter (D), a wall thickness (t) and being subjected to an internal pressure (p) is conventionally defined as follows:

$$U = \frac{p\,(D - 2t)}{2t} \qquad (a)$$

However, more accurate results are obtained by using the formula for thick-walled tubing:

$$U = \frac{p}{\log_e \dfrac{D}{D - 2t}} \qquad (b)$$

assuming that the maximum value of tension occurring at the outside surface is deciding. For thin-walled tubing this equation can be approximated by a simpler formula:

$$U = \frac{p\,(D - t)}{2t} \qquad (c)$$

which assumes linear, but not uniform, stress distribution in the wall such that the difference between the stress at the inside and

outside surface is equal to the internal pressure. Neither (b) nor (c) takes into account the additional longitudinal stress, and because of this, the values obtained should be, and are, on the average somewhat higher than those obtained from a tensile test (1).

Values of per cent elongation were taken for small elongations from the stress-strain curves, Fig. 4; but elongation values for large elongations were found to be more reliable if determined from measurements of the circumference after fracture. A check on the

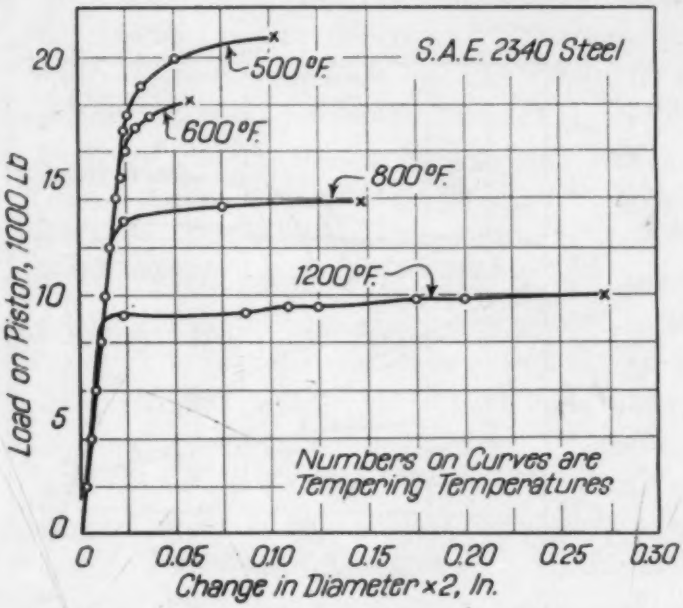


Fig. 4—Pressure Change in Diameter Curves for Thin-Walled Tubing Under Internal Pressure and Longitudinal Tension.

elongation was obtained by measuring the thickness at numerous points after failure. This reduction was uniform over the whole circumference for specimens having less than 5 per cent elongation; and because of the constancy of length, or zero strain in the longitudinal direction, the elongation (e) and average wall thickness reduction (Q) are then interrelated by the following equation:

$$e = \frac{Q}{1 - Q}. \qquad (d)$$

In the more ductile specimens, the wall thickness reduction was found to be uniform over the major portion of the circumference, while local necking occurred at the failure and sometimes a minor necking occurred at another point also. The average of the wall thickness reductions again corresponded to the elongation.

The maximum reduction in wall thickness at the break corresponds to the contraction in area of a tensile test, while the uniform reduction in wall thickness corresponds to the uniform reduction in cross-sectional area of a tensile bar. This uniform reduction of the more ductile specimens was found to be consistently between 3 and 4 per cent, or approximately the same as that of the cylindrical tensile test bars. Also because of the generally small values of contraction in area, the elongation of the tubing is not very different from the uniform reduction.

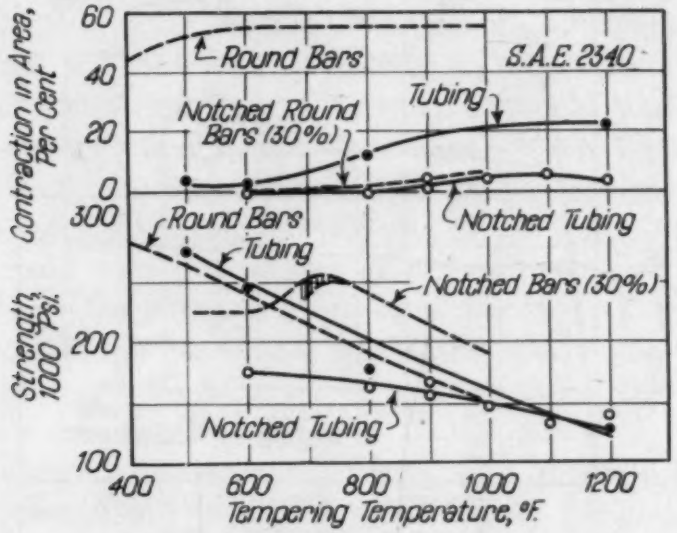


Fig. 5—Strength and Ductility of Unnotched and Notched Tubing.

The failures obtained with unnotched specimens were usually of the shear type and occasionally of the cup and cone type. Some necking was apparent even in the least ductile specimens.

The stress-strain curves for the unnotched tubing, Fig. 4, illustrate the effect of the tempering temperature on the various tensile test characteristics of the steel tubing. The circumferential ultimate strength and contraction in area are represented in Fig. 5 together with the corresponding properties obtained on cylindrical tensile test bars. For purposes of comparison, the results for notched tubing and notched tensile test bars, as discussed later, have been represented in Fig. 5 also. Due to the limited number of tests on the tubing and the scattering of the results, the trend of the different properties is not so definite as with cylindrical bars. The scattering may be explained by various factors, such as the uncertainty regarding the

actual dimensions at the break, or the presence of slight decarburization or other variations in chemical composition. These factors, of course, are more effective in the testing of thin-walled tubing than in the testing of cylindrical bars.

The values of ultimate strength for the tubing agree within approximately ± 5 per cent with the more accurate values of ultimate strength for rod heat treated in the same manner. The average of the values for tubing is somewhat higher, almost 5 per cent, than that for the cylindrical bars, and this corresponds to modern conceptions on the effect of a triaxial stress on the yield strength and ultimate strength of ductile metals. In fact, it was expected that this difference would amount to approximately 10 per cent (1).

On the contrary, the ductility of the tubing as represented by the values of contraction in area is in all cases only a fraction of that of cylindrical test bars. This is particularly true for the hard tempers, which show contractions of only a few per cent, while identically treated cylindrical bars may possess contractions of more than 50 per cent. The discrepancy is less pronounced at the lower strength levels of 150,000 pounds per square inch, where a contraction in area of over 20 per cent was observed for the tubing.

Thus, there is a considerable difference in ductility of a heat treated steel depending upon whether it is subjected to uniaxial tension (in the shape of a cylindrical rod) or to biaxial tension (in the shape of tubing) in such a direction that failure will occur in the circumferential direction. The dependence of the ductility upon the tempering temperature under biaxial tension (tubing) appears to be much the same as in triaxial tension, the triaxiality being of a low magnitude, such as that represented in a test on a cylindrical bar provided with a shallow (5 to 10 per cent) notch.

Another factor which differs for rod and tubing and which might be made responsible for the lower ductility of the tubing is the direction of fiber. While the fiber of a rod can be compared to a bundle of wires, a tube (and similarly a strip) can be visualized as being composed of flat leaves. The "transverse" direction of inferior strength is any lateral direction of the rod, but only the radial direction, or direction perpendicular to the surface in the case of tubing. Therefore, the properties in the circumferential direction of tubing should be intermediate between the longitudinal and transverse properties, and not radically different from the longitudinal properties. This has been confirmed by some tests in this respect,

(3), (4) and also by some observations regarding the tensile properties of forgings (5).

Bursting Tests on Notched Tubing

The test procedure for notched tubing was essentially the same as for unnotched specimens.

For the purpose of measuring the depth of the notch before the test, a pair of micrometers was fitted with a chisel-shaped anvil having an included angle of approximately 40 degrees. Diameter readings were taken with the anvil placed in the notch and also adjacent to the notch on each side. The depth of the notch was taken as the average of the readings on each side of the notch minus the reading taken with the anvil inserted in the notch. The final value for notch depth was taken as the average of ten measurements made as above and taken at various points along the length of the notch. For the purpose of computing notch strength, the depth of the notch was subtracted from the wall thickness measured only at the notch, rather than the average wall thickness around the circumference.

For all the notched tubes, the stress-strain (load diameter change) curves consisted only of a straight line up to the breaking point. Thus, all deformations in the tube were elastic except for small plastic movements in the immediate vicinity of the notch, and as a result, the per cent elongation for all of the notched tubes was zero.

Because of the small elastic deflections of the notched tubes, it was found difficult to produce a longitudinal stress in the tubes by the method used in the case of the unnotched tubes. In order to secure the desired longitudinal stress before fracture, it would be necessary to reduce the clearance between the protection tube and the shoulders on the cap and adaptor to a value below 0.001 in., and this would cause considerable mechanical difficulties. Consequently no shims were used, and only that longitudinal stress was present which was due to the tendency of the notched section to contract longitudinally and the restraining effect of the thicker material adjacent to the notch. Probably the longitudinal tension due to this effect is comparable to that produced in the unnotched specimens because of the same condition of zero longitudinal strain.

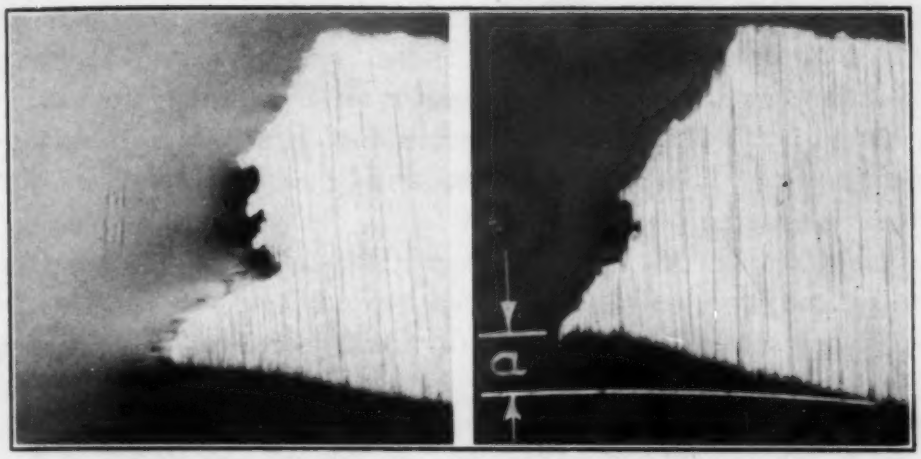


Fig. 6—Failure of Notched S.A.E. 4140 Tubing Quenched from 1550 Degrees Fahr. Tempered at 1000 Degrees Fahr. × 25.
Fig. 7—Failure of Notched S.A.E. 4140 Tubing Quenched from 1550 Degrees Fahr. Tempered at 1200 Degrees Fahr. × 25.

As "notch strength" (U_n), the average ultimate strength of the notched section is introduced. This is determined by the following equation:

$$U_n = p\left[\frac{D-2t}{2t_n} + \tfrac{1}{2}\right] \qquad (e)$$

where p is the internal pressure, D is the outside diameter, t is the thickness of the unnotched section, and t_n is the thickness of the notched section.

The types of failure in notched tubing are illustrated in Figs. 6 and 7 by sections perpendicular to the axis of the tube. It was found that for the softer tempers, some contraction in area occurred at the notch. This quantity was calculated from measurements made with a micrometer microscope on a section of the tube perpendicular to the axis, Fig. 7. If it is assumed that no plastic flow occurs except in the immediate vicinity of the notch, the original wall thickness can be taken as the wall thickness near the notch after fracture. Then the change in wall thickness ("a" of Fig. 7) can be measured directly as the radial distance from the inside surface of the tube directly under the notch to the surface of the tube near the notch. Such measurements at different points along the fracture and on opposite sides of the fracture of any one specimen agreed to within ± 0.0002 inch, or ± 10 per cent of their value. These values cannot be considered as very reliable but they illustrate fairly accurately the differences in ductility for various tempering temperatures.

Thus, the notched tubular specimens yielded two characteristics, the average ultimate strength or "notch strength" and the decrease in thickness after failure which is designated as "contraction in area" or "ductility". These are included in Fig. 5, which also shows the corresponding values for cylindrical tensile test bars provided with a 30 per cent notch (6).

The trend of the notch strength curve for various tempers of heat treated steel tubing differs considerably from that for cylindrical bars. There appears to be a gradual decrease of notch strength

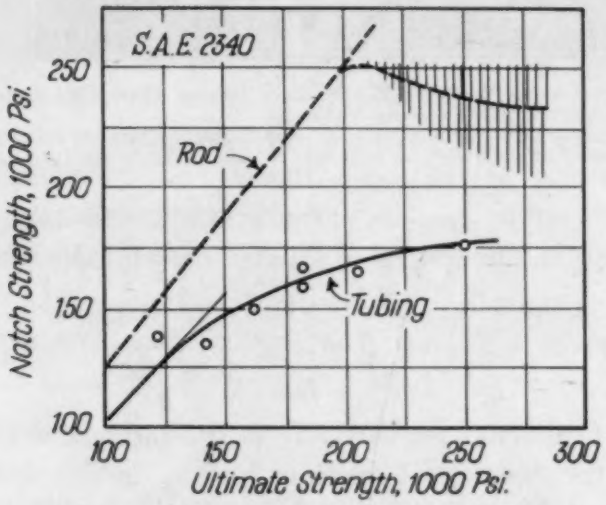


Fig. 8—Relation Between Notch Strength and Ultimate Strength of Tubing.

with increasing tempering temperature, Fig. 5, or with decreasing hardness and ultimate strength throughout the whole range of commercial tempers, Figs. 5 and 8. In the lower strength levels, below 155,000 pounds per square inch approximately, the notch strength is practically the same as the ultimate strength of the tubing. In this range of strength, the contraction in area is 3 per cent or higher, indicating a ductile condition of the steel. With increasing ultimate strength, the notched specimens gradually become more brittle, and the contraction in area becomes practically zero at a strength level of approximately 190,000 pounds per square inch. At this point, the notch strength is approximately 90 per cent of the ultimate strength or close to the yield strength of the tubular specimens. At still higher strength levels, the notch strength becomes considerably lower than the ultimate strength of unnotched specimens, the minimum ratio being approximately 70 per cent.

The following differences are apparent from Figs. 5 and 8 regarding the notch strengths of tubular and cylindrical specimens: (a) The values obtained on tubing in the brittle range scatter considerably less for tubing than for cylindrical specimens. This can be explained by the fact that the strain in tubing is symmetrical by the nature of the loading while the problem of centering circular tensile test specimens has not been solved as yet. Thus, additional bending strain originating from eccentricity and causing excessively low values of notch strength should be absent in tubing. (b) In tubing, the brittle range extends to considerably lower ultimate strength levels than in cylindrical bars. Steels having an ultimate strength as low as 150,000 pounds per square inch have a low ductility if tested as notched tubing.

In considering these results, it must be kept in mind that the severity of the type of notch investigated in the tubing is difficult to estimate, but the notch is probably not particularly radical. However, it is difficult to produce sharper notching, e.g., notches on both the outer and inner surfaces. The stress state in notched tubing is generally not known, but the small difference between the notch strength of ductile tubing and the ultimate strength of unnotched tubing indicates that the average value of the radial stress which retards the plastic flow is small at the moment of failure. On the other hand, a substantial circumferential and radial stress concentration may exist in a small area, thus accounting for the brittle behavior of otherwise ductile steels.

The testing of tubing yields the conclusion that the combination of conditions present in notched tubing favors the embrittlement of otherwise ductile steels to a higher extent than any other set of conditions which has been investigated up to the present time. High grade alloy steels, such as S.A.E. 2340, become noticeably brittle if they are heat treated to a strength level of 150,000 pounds per square inch or higher and subjected to internal pressure when in the form of notched tubing.

Tubing Provided With a Double Notch

From the previously discussed tests on tubing notched on the outside surface only, the conclusion was drawn that such a notch constitutes a powerful embrittling factor. The circumferential strength of the notched section was found to be identical with the

ultimate circumferential strength of unnotched tubing up to a strength level of 150,000 pounds per square inch. At higher strength levels, however, the notch strength was less than the ultimate strength, to an extent increasing with increasing strength level, indicative of a corresponding increase in notch brittleness.

On the other hand, it was surprising that such a notch did not cause an increase in strength for the ductile condition. Such an increase should be expected from the presence of an average transverse (radial) stress, which is considered as one of the decisive factors regarding the embrittlement of steels.

Therefore, both an increase in notch strength of the lower strength levels and a decrease in strength and/or ductility for the higher strength levels were expected for specimens provided with a double notch.

Considerable inaccuracy was unavoidable due to the difficulties of machining such notches. The two notches are quite likely to be not exactly opposite each other. Furthermore a slight error in the depth of the notch causes a considerable error in the per cent notch depth when the wall is quite thin. For the purpose of comparison it was intended to have 30 per cent of the thickness or cross-sectional area removed by notching for each of the notched tubes (15 per cent by each notch for the double notches). Actually, this "notch depth" varied from 23 to 52 per cent for all the tests made on tubing. In spite of the expected difficulties, the notches were machined with as great care as possible, with the hope that the effects would be large in comparison to the inaccuracies.

The preparation and testing procedure for the specimens with double notches were identical with the previously described procedure except for the following items. The outside notch was made with a milling cutter, and the inside notch was made using a boring bar mounted on the carriage of a lathe. The thickness of the section remaining after notching was measured by means of a large micrometer with chisel-shaped anvils on both ends. The anvil on the spindle end had to be provided with an accurate seat, so that the spindle could rotate in the anvil.

The results of the tests are tabulated in Table I and plotted in Fig. 9 (along with previous results on tubing).

It can be concluded from these results that tubing with a double notch does not show any greater tendency toward embrittlement than tubing with a notch on one side only.

Table I

S.A.E. No.	Tempering Temp. (½ hr.) Degrees Fahr.	Notch Strength Pounds Per Square Inch	Notch Depth Per Cent
2340	800	216,000	38
2340	1200	152,000	37

The notch strength values, however, are considerably higher for tubing provided with a double notch than those for single notch tubing.

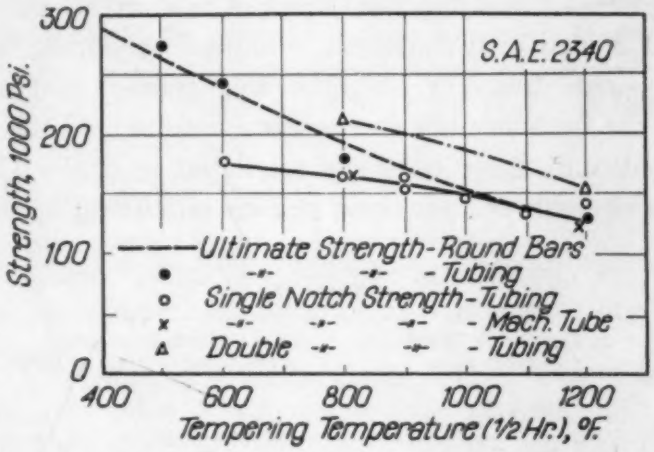


Fig. 9—Bursting Strength of Tubing Provided With a Double Notch and of Notched Tubing Machined from Rod.

Thus it can be concluded (a) that a double notch is not a more severe embrittling factor than a single notch, but (b) that the strength-increasing effect, which is predominant in the ductile condition, only occurs when the notch is on both sides.

Tubular Specimens Machined From Rod

As discussed, the embrittlement of tubing in the circumferential direction by the presence of a longitudinal notch (or a longitudinal double notch) is much more pronounced than the embrittlement of rod in the longitudinal direction by the presence of a circumferential notch. While the tensile strength of rod is not decreased by notching unless the strength level of the steel is materially higher than 200,000 pounds per square inch, the bursting strength of tubing is adversely affected by notching, when the strength level is raised above a value of 150,000 pounds per square inch.

A possible explanation for this different behavior is offered by the different fiber structure of rod and tubing. The longitudinal direction of rod is the direction of fiber, which is generally characterized by strength properties superior to those of any other direction. On the contrary, the circumferential direction of tubing is intermediate between the longitudinal, or fiber direction, and the principal transverse (or radial) direction, which presumably should possess particularly low strength properties.

In order to investigate the validity of these assumptions a few tests were made on tubular specimens, 1.375 inches in outside diameter and 0.063 inch wall thickness, obtained by boring the center out of solid 1½-inch diameter rod otherwise treated and provided with a notch on the outside only in the same manner as the tubing.

The results of these tests are tabulated in Table II and plotted in Fig. 9 along with the previous results on tubing.

Table II

S.A.E. No.	Tempering Temp. Degrees Fahr.	Notch Strength Pounds Per Square Inch	Notch Depth Per Cent
2340	800	164,000	33
2340	1200	126,000	52

The notch strength values determined for the tubular specimens machined from rod closely agree with those of tubing, other conditions being identical. It can be definitely concluded from these results that the notch sensitivity in the transverse direction of rod is no greater than that in the circumferential direction of tubing. No explanation can be given for this surprising result.

Bibliography

1. G. Sachs, "Some Fundamentals of the Flow and Rupture of Metals," *Transactions,* American Institute of Mining and Metallurgical Engineers, Institute of Metals Division, 1941, p. 12-29.
2. M. Gensamer, "Strength of Metals Under Combined Stresses," Published by the American Society for Metals, Cleveland, 1941.
3. A. F. Maier, "Effect of Stress State on Ductility of Metallic Materials," *Mitt. Materialpruefungs Anst.,* Stuttgart (1935).
4. E. Siebel and A. Maier, "Effect of Multiaxial Stress States on Ductility," *Zeit. V. D. Ing.,* Vol. 77, 1933, p. 1345-1349.
5. G. Sachs, "Some Observations on the Forging of Strong Aluminum Alloys," *Journal,* Institute of Metals (London), Vol. 64, 1939, p. 261-283.
6. G. Sachs and J. D. Lubahn, "Notched Bar Tensile Tests on Heat Treated Low Alloy Steels," American Society for Metals Convention, 1942.
7. G. Sachs and J. D. Lubahn, "Effects of Notching on Strained Metals," *Iron Age,* Vol. 150, 1942, Oct. 8, p. 31-38 and Oct. 15, p. 48-52.

ORAL DISCUSSION

A. V. DE FOREST[1]: The study of ductility as influenced by restraint caused by combined stress is important, especially in many welding applications.

As we all know, ductility in the longitudinal direction is by no means sufficient to define how the material is going to behave under biaxial conditions, and yet the study of such conditions lag greatly, so this contribution is an important one.

The use of the tubing is, of course, the simplest one from the mechanical point of view. On the other hand, it is also a very difficult experimental technique; one of the difficulties is illustrated in the notch on the outside of the tube where quite evidently combined bending and circumferential tension emphasizes the notch effect of the single notch as compared with the double notch, which if it could be formed perfectly would give a balanced tension across the material at the root of the notch. That is a difficult thing to do but these authors are used to doing difficult things.

Personally, I do not totally like the word "embrittlement" as applied to these notch effects. I think it is much fairer to regard the notch as restraining the ductility in shorter gage lengths, so that perhaps restraint of ductility rather than embrittling effect would be a nice way of explaining it. In general, the word embrittle means something like grain growth or grain boundary attack or some metallurgical change rather than the pure mechanical effect which we are dealing with in the case of notches and in which the material has remained totally unchanged.

In the case of the practical method of testing such conditions it may be hoped that these experiments on tubing, which are fairly determinate mechanically, can some day be translated back into some form of circumferential notch and longitudinal testing such as those proposed by Professor Hague in England with a very severe and extremely accurately determined notch condition leading to triaxial stress.

DANIEL ROSENTHAL:[2] There is one question I should like to ask in connection with this test. What would be, in the opinion of the authors, the behavior of a cast iron? In other words, if we make a deep notch in a cast iron, how will the ultimate stress be affected by this notch? The reason why I bring this question out is that some people think that the behavior under a triaxial constraint caused by a notch can tell us how the material will behave in welding. Now, cast iron is most difficult to weld. Cracks during welding are likely to occur under almost any conditions, except possibly when very carefully preheated. If a correlation between this behavior and the results of a notch test can be established, very useful information indeed will be available to the welders.

Authors' Reply

Professor de Forest suggested that the word embrittlement as applied to the loss of ductility by notching should be replaced by the term "restraint of

[1]Professor of mechanical engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[2]Assistant professor of metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

ductility". This is partly true in that the strain distribution in any possible direction of measurement is not uniform, and consequently the average strain at fracture, measured over some finite gage length, will not be a true measure of the ductility, or the maximum strain possible without failure.

However, this conception is not sufficient to explain all of the embrittlement due to notching. Notching causes not only a stress concentration resulting in a non-uniform strain distribution, but it also causes a triaxial stress state. This in itself is sufficient to cause a loss of ductility, even though the strain distribution is uniform, as can be shown by tests on unnotched tubing, see Fig. 5, and also reference 4.

Regarding Professor Rosenthal's question as to the effect of notching on gray iron, it was found by Kuntze, see Fig. 6 of reference 7, that the notch strength is nearly independent of the notch depth. In other words, notching has practically no effect on gray iron at all. The explanation of this surprising fact may be that cast iron has a large number of very sharp internal notches due to the presence of the graphite flakes, and the addition of an external notch would therefore produce an insignificant additional effect on the behavior of the metal.

THE TANTALUM-CARBON SYSTEM

By Finley H. Ellinger

Abstract

A tentative constitutional diagram of the tantalum-rich end of the tantalum-carbon system has been constructed from metallographic, X-ray and melting point data.

Tantalum forms two carbides: Ta_2C (3.2 per cent carbon) and TaC (6.2 per cent carbon). TaC will dissolve up to 1.2 per cent tantalum at 3400 degrees Cent. (6150 degrees Fahr.).

Two eutectics are present in this system. The Ta-Ta_2C eutectic occurs at 0.6 per cent carbon and 2800 degrees Cent. (5070 degrees Fahr.). The TaC-graphite eutectic occurs at approximately 10 per cent carbon and 3300 degrees Cent. (5970 degrees Fahr.).

MOST of the previous work on alloys of tantalum and carbon concerns the preparation and properties of the tantalum carbide, TaC, of 6.2 per cent carbon content. This carbide is most commonly prepared by the dissociation of a tantalum compound, usually Ta_2O_5, with carbon at some elevated temperature. The melting point of TaC has been placed at about 4100 degrees absolute by Friederich and Sittig (1)[1] from the results of 18 determinations under various conditions. Agte and Alterthum (2) found the melting point at 4150 $\pm$ 150 degrees absolute which is in good agreement with the work of Friederich and Sittig.

The existence of a lower carbide has been reported by Burgers and Basart (3). They identified it as being Ta_2C of 3.2 per cent carbon content and crystallizing in a close-packed hexagonal lattice. Some evidence of the occurrence of two modifications of Ta_2C was also offered.

Since TaC is an important constituent of some cemented carbide tool materials, an investigation of the tantalum-carbon system seemed

[1]The figures appearing in parentheses refer to the bibliography appended to this paper.

A paper presented before the Twenty-fourth Annual Convention of the Society held in Cleveland, October 12 to 16, 1942. The author, Finley H. Ellinger, is associated with the Cleveland Wire Works, Lamp Department, General Electric Co., Cleveland. Manuscript received June 1, 1942.

advisable as an aid to a better understanding of the sintered carbide structures.

Materials and Apparatus

The tantalum used in this investigation was in the form of 200-mesh powder of better than 99.9 per cent purity. Alloys of the desired compositions were prepared by briquetting intimate mixtures of tantalum and graphite, or of tantalum and tantalum-carbon alloy powders of various carbon contents. The most successful method of producing these alloy powders was to make a mixture of tantalum and graphite of the desired composition, press into a bar, sinter in vacuum, usually at 2400 to 2500 degrees Cent. (4350 to 4530 degrees Fahr.), and then crush into 200-mesh powder. This powder was then analyzed for carbon content since it was always higher than the calculated value due to absorption of carbon from the atmosphere during sintering.

Attempts to form the intermediate carbide Ta_2C by heating Ta_2O_5 in the presence of the requisite amount of carbon, in the manner in which TaC is manufactured, were not successful. Instead of the carbon replacing all of the oxygen of the oxide to form Ta_2C, the resultant product was a mixture of TaC and tantalum oxide.

For sintering and melting of the tantalum-carbon pressed bars, two types of equipment were used. One of these was a carbon resistor furnace, the carbon crucible itself acting as the resistor and being enclosed in a partial vacuum. Temperatures up to 3400 degrees Cent. (6150 degrees Fahr.) were obtainable with this furnace. It has been described more fully by W. P. Sykes (4).

An Ajax-Northrup high frequency vacuum furnace was found to be well adapted for sintering tantalum-carbon alloys, but not as high temperatures could be obtained as in the resistor furnace. However, this furnace was suitable for continuous operation at temperatures up to 2500 degrees Cent. (4530 degrees Fahr.). Higher temperatures could be used for short periods of time. Temperature measurements were made by the calibration of power input and time against the melting points of the Mo-Mo_2C eutectic (2200 degrees Cent.), the W-W_2C eutectic (2475 degrees Cent.) and tantalum (2850 degrees Cent.).

On operation of the furnace it was first evacuated to 25-50 microns Hg. After the power had been applied, however, and the

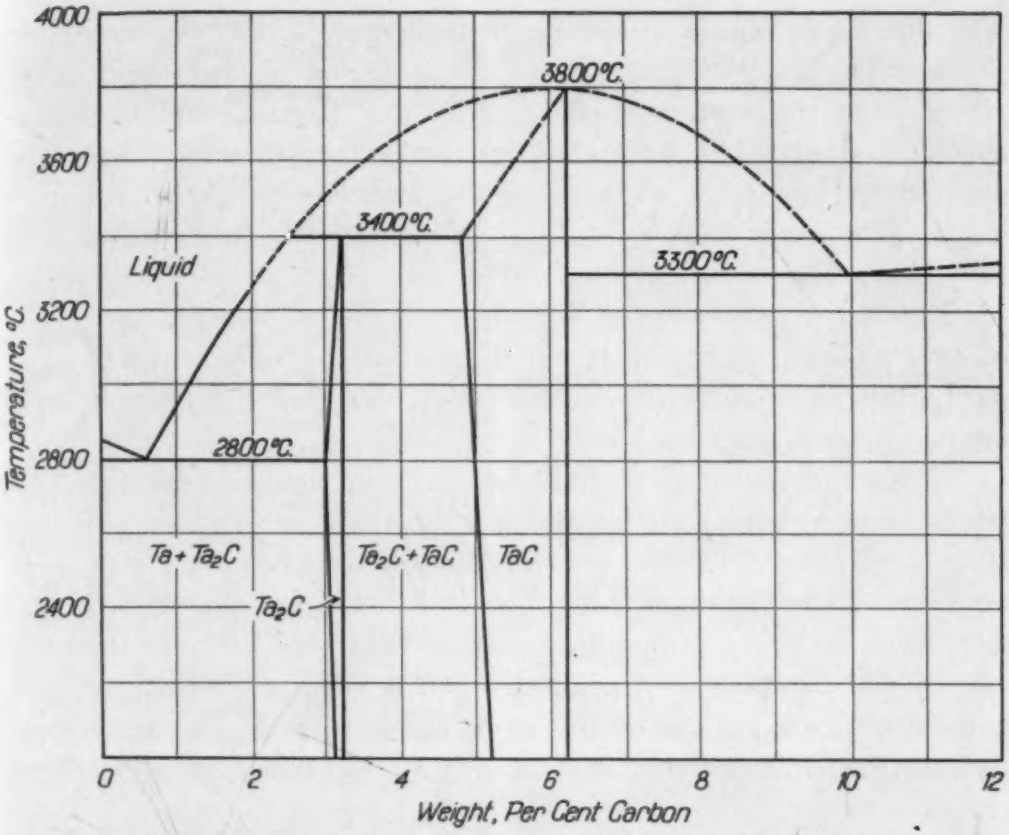


Fig. 1—Tentative Constitutional Diagram of Tantalum-Rich End of Tantalum-Carbon System.

temperature rose above 2000 degrees Cent. (3630 degrees Fahr.), the vacuum would fall to 1 to 3 millimeters Hg. This decrease was attributed to the carbon vapor formed from the carbon crucible at the high temperature and low pressure at which the furnace was being used.

Constitutional Diagram

The tentative constitutional diagram of the tantalum-rich end of the tantalum-carbon system is shown in Fig. 1 as constructed from metallographic, X-ray and melting point data.

Preliminary evidence of the formation of tantalum carbides was obtained by carburization of a piece of tantalum rod in the Ajax vacuum furnace at 2300 to 2400 degrees Cent. (4170 to 4350 degrees Fahr.) for ½ hour. The carbides which formed on the surface of the rod are shown in Fig. 2. This carburized zone is made up of two distinct shells, each of which, in turn, is composed of two layers.

The outermost region of the outer shell may be seen to consist of polyhedral grains of TaC which change from a yellowish tint to white from the surface inward or in the direction of decreasing carbon content. The white TaC gradually merges into a region of lamellar grains which are made up of alternate plates of TaC and Ta_2C. The inner shell, solid Ta_2C, is separated from the outer shell by a sharp line of demarcation. The solid Ta_2C merges into grains of Ta_2C which are striated with layers of Ta, probably the result of precipitation on cooling from the carburizing temperature. The dark gray inner core of tantalum is spotted with islands of Ta_2C next to the carburized surface.

Sintered Alloys—Sintered structures of tantalum-carbon compositions of less than 3.2 per cent carbon show two phases, tantalum and Ta_2C (Fig. 3). No appreciable solid solubility of carbon in tantalum could be detected. The Ta_2C grains are readily distinguished from the tantalum matrix both by their greater hardness and by the striations of tantalum which develop within them. The amount of Ta_2C present in the structure increases with carbon content until at 2.9 per cent carbon only a slight broken network of tantalum remains (Fig. 4).

Since the intergranular tantalum is absent when the carbon content of the Ta_2C-Ta alloys reaches 3.0 per cent, it was concluded that Ta_2C (3.2 per cent carbon) will dissolve 0.2 per cent tantalum in solid solution at the eutectic temperature and that the striations of tantalum in the Ta_2C grains are the result of precipitation on cooling from the sintering temperature.

An alloy of 3.2 per cent carbon, the stoichiometric composition of Ta_2C, no longer shows the striations of tantalum, but is made up entirely of a single phase as shown in the characteristically porous structure of Fig. 5. Irregular markings which develop on etching are characteristic of this phase. Fig. 6 shows that these markings are also present in the Ta_2C grains which are striated with plates of tantalum.

As the carbon content of the alloys is increased above 3.2 per cent carbon another phase appears which was identified as TaC. Fig. 7 shows the structure of a 3.5 per cent carbon alloy which has been heated to 3300 degrees Cent. (5970 degrees Fahr.). This structure shows massive areas of Ta_2C and areas composed of Ta_2C and TaC. Fig. 8 shows clearly the continuity of the bands of Ta_2C

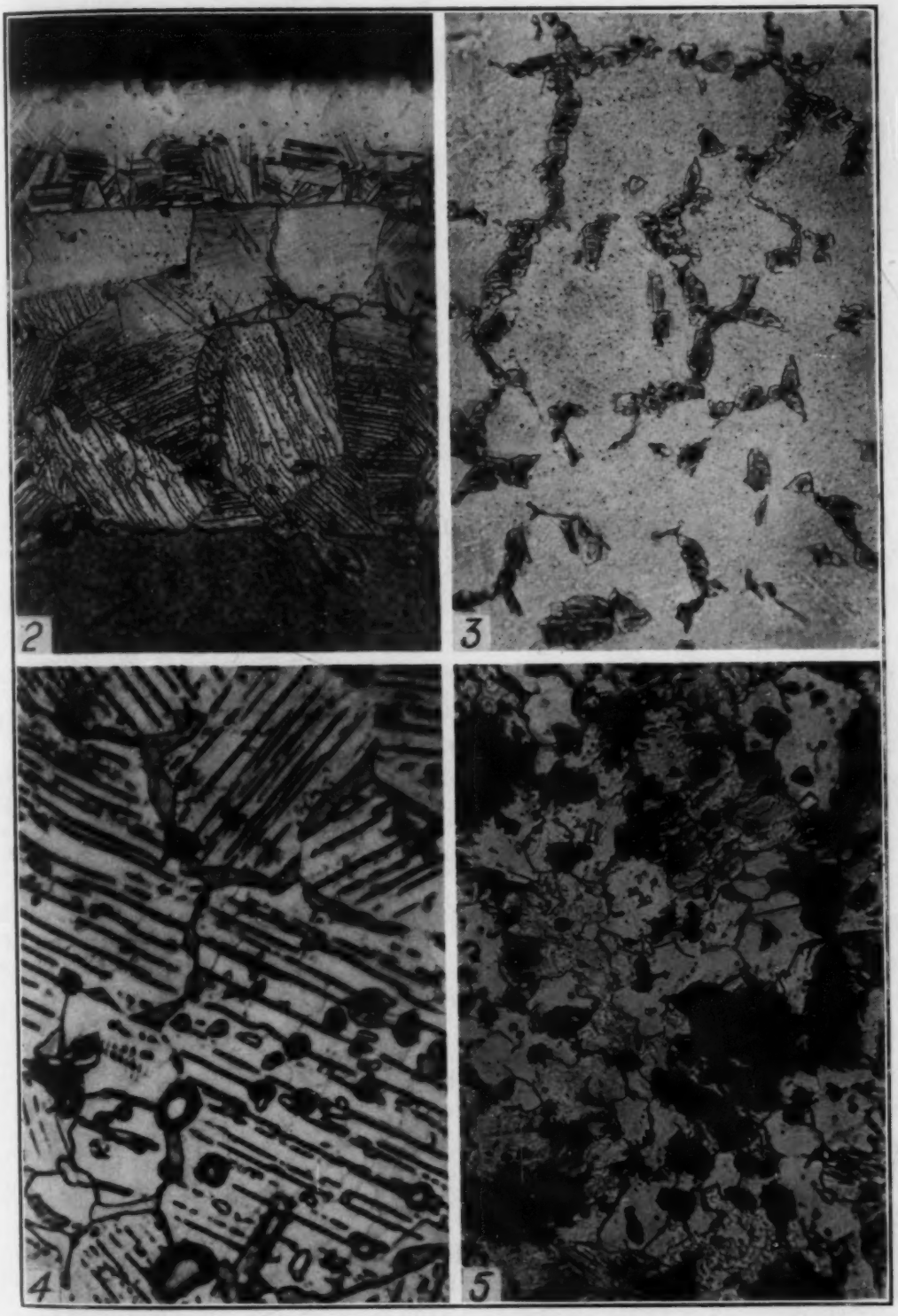


Fig. 2—Section Through Carburized Tantalum Rod Showing Shells of TaC and Ta_2C. × 200.

Fig. 3—Tantalum Plus 0.6 Per Cent Carbon. Sintered 2500 to 2700 Degrees Cent. —5 Minutes. Ta Plus Ta_2C. × 200.

Fig. 4—Tantalum Plus 2.9 Per Cent Carbon. Sintered 2400 Degrees Cent.—30 Minutes. Ta_2C Plus Ta. × 1500.

Fig. 5—Tantalum Plus 3.2 Per Cent Carbon. Sintered 3000-3300 Degrees Cent. —1 Minute. Massive Ta_2C. × 200.

with the massive Ta_2C. With increasing carbon content, the massive Ta_2C diminishes and the structure consists entirely of the mixture of Ta_2C and TaC as seen in Figs. 9 and 10, the proportion of TaC increasing with carbon content. At 5 per cent carbon the striated structure is no longer present and the alloy now consists of polyhedral grains of TaC (Fig. 11). As the carbon content is increased to 6.2 per cent, the TaC grains gradually change from white to the golden yellow tint characteristic of TaC, but at 5.5 per cent carbon the yellowish tint is still difficult to detect.

Fused Alloys—Sintered structures consisting of tantalum plus Ta_2C were found to fuse or partially fuse at 2800 degrees Cent. (5070 degrees Fahr.). Fusion was most complete in the alloys of less than 1 per cent carbon. Examination of the fused structures showed the presence of a eutectic at some composition of less than 1 per cent carbon and an alloy of 0.6 per cent carbon appeared most nearly to be 100 per cent eutectic (Fig. 12).

During the course of fusing these low carbon compositions it was noted that the eutectic temperature was very close to the melting point of tantalum (2850 degrees Cent.),, in fact, it was difficult to distinguish at all since the difference was within the degree of accuracy of the temperature measurements which was about $\pm$ 50 degrees Cent. However, the eutectic temperature was more firmly established by heating to 2800 degrees Cent. (5070 degrees Fahr.) a section of tantalum rod which had been slightly carburized on the surface. Fig. 13 shows that the surface of the rod has fused and has crystallized in the typical Ta-Ta_2C eutectic structure. The tantalum core with some particles of Ta_2C has not fused, as has neither the thin outermost shell of Ta_2C.

During the treatment of the alloys a shell of carbide always formed on the surface of the specimens. In most cases, if the alloy was not heated much above 2800 degrees Cent. (5070 degrees Fahr.) the shell would remain unfused and it was this condition which allowed the structures of Ta_2C and Ta to be preserved for metallographic examination. By allowing a minimum of time for the interior of the specimen to be molten, the shell would usually remain intact and thus act as a crucible for the liquid metal, preventing it from coming in contact with the carbon crucible and thus rapidly absorbing carbon.

An alloy of 1 per cent carbon heated to above the eutectic tem-

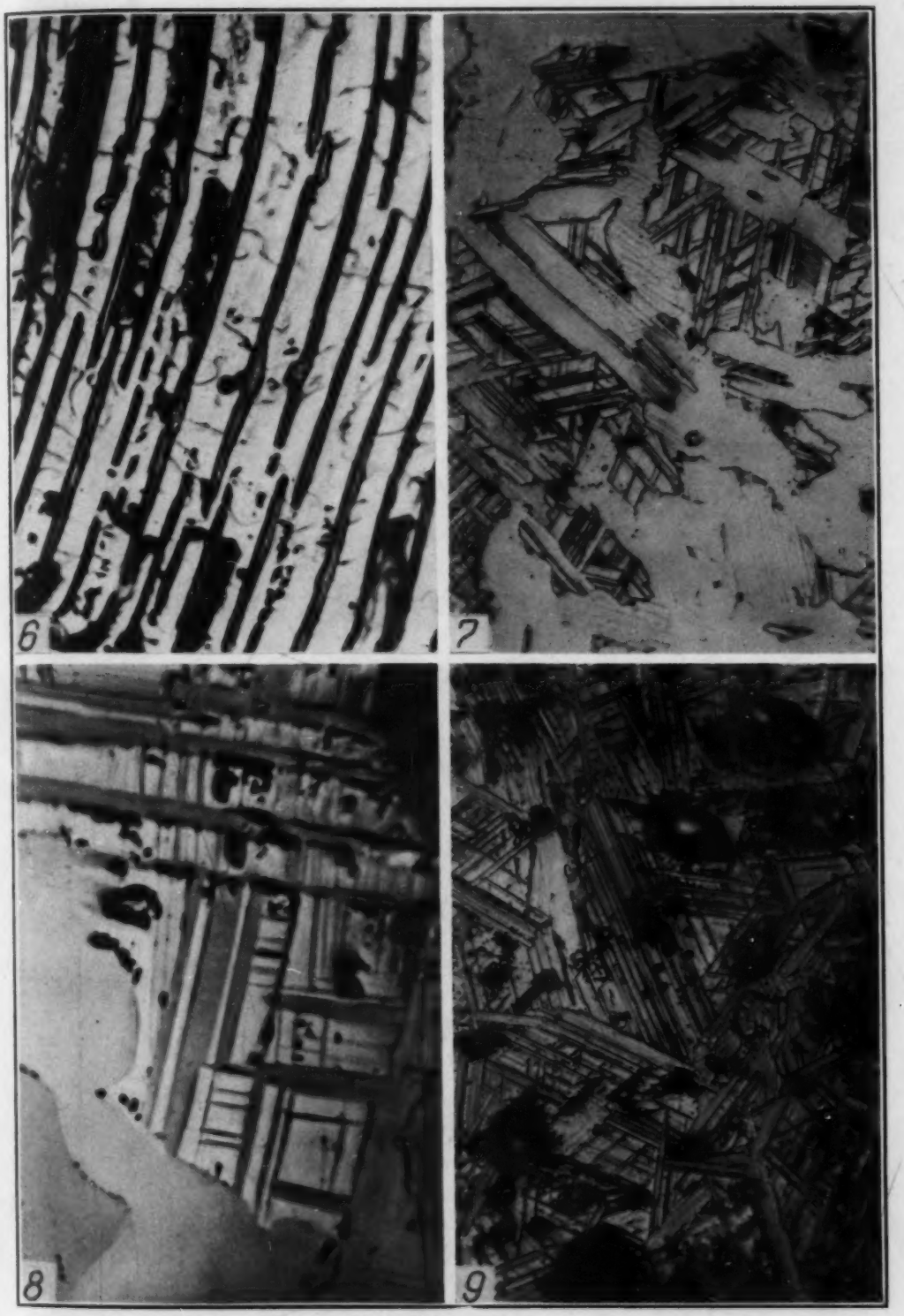


Fig. 6—Tantalum Plus 3.0 Per Cent Carbon. Sintered 2400 Degrees Cent.—30 Minutes and 2800 Degrees Cent.—15 Minutes. Ta_2C + Ta. × 1500.

Fig. 7—Tantalum Plus 3.5 Per Cent Carbon. Sintered 2500 Degrees Cent.—1 Minute and 3300 Degrees Cent.—½ Minute. Ta_2C + TaC. × 200.

Fig. 8—Tantalum Plus 3.0 Per Cent Carbon. Sintered 2000 Degrees Cent.—2 Minutes and 3200 Degrees Cent.—½ Minute. Micro from Shell of Specimen. Ta_2C + TaC. Unetched. × 1500.

Fig. 9—Tantalum Plus 4.0 Per Cent Carbon. Sintered 2000 Degrees Cent.—2 Minutes and 3300 Degrees Cent.—½ Minute. Ta_2C + TaC. × 500.

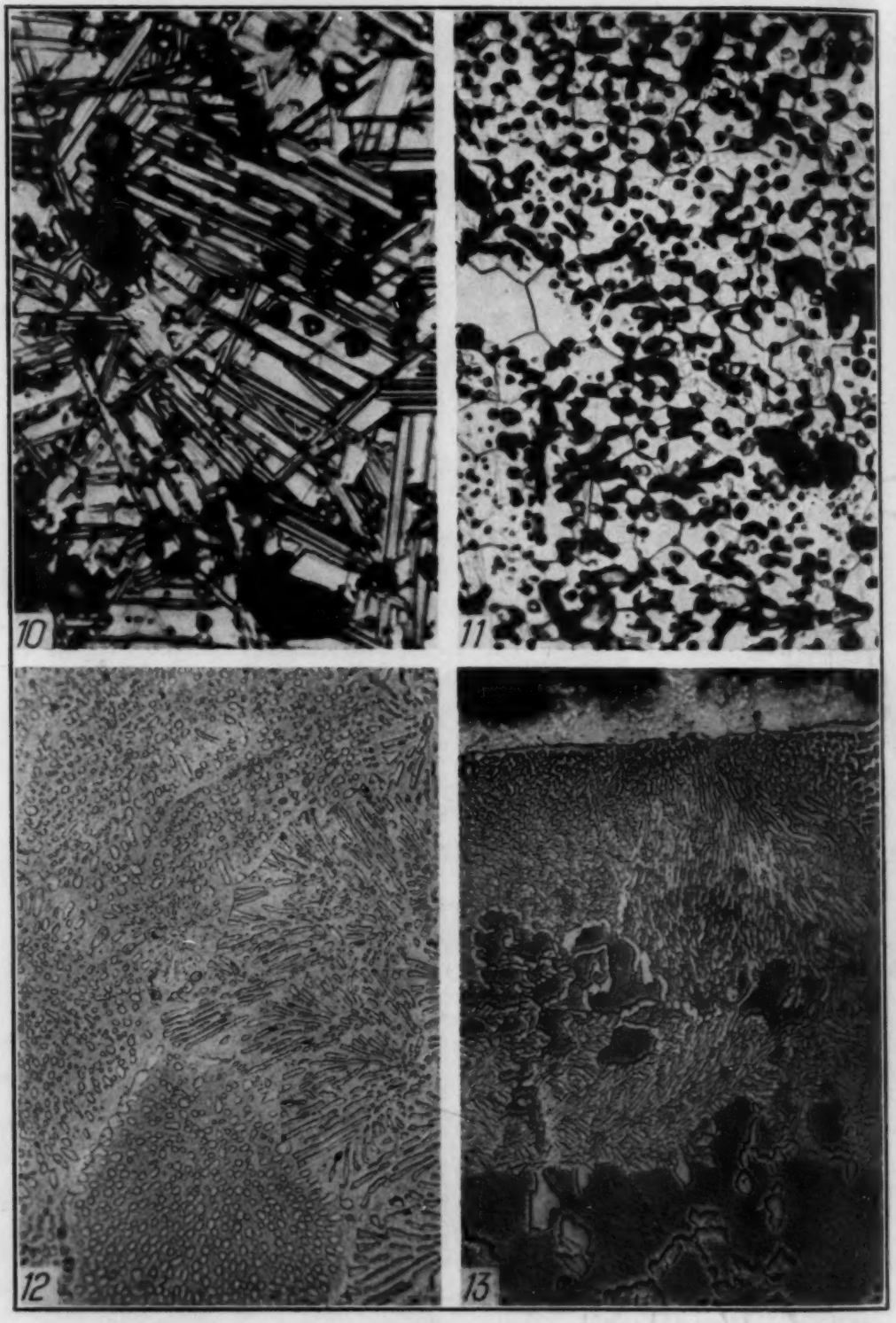


Fig. 10—Tantalum Plus 4.5 Per Cent Carbon. Sintered 2500 Degrees Cent.—1 Minute and 3350 Degrees Cent.—½ Minute. Ta_2C + TaC. × 1500.

Fig. 11—Tantalum Plus 5.6 Per Cent Carbon. Sintered 3300 Degrees Cent.—½ Minute. Massive TaC. × 500.

Fig. 12—Tantalum Plus 0.6 Per Cent Carbon. Fused at 2800 Degrees Cent. Ta-Ta_2C Eutectic. × 200.

Fig. 13—Tantalum Rod, Carburized and Heated to 2800 Degrees Cent. Typical Eutectic Structure Formed on Surface of Specimen. Unetched. × 100.

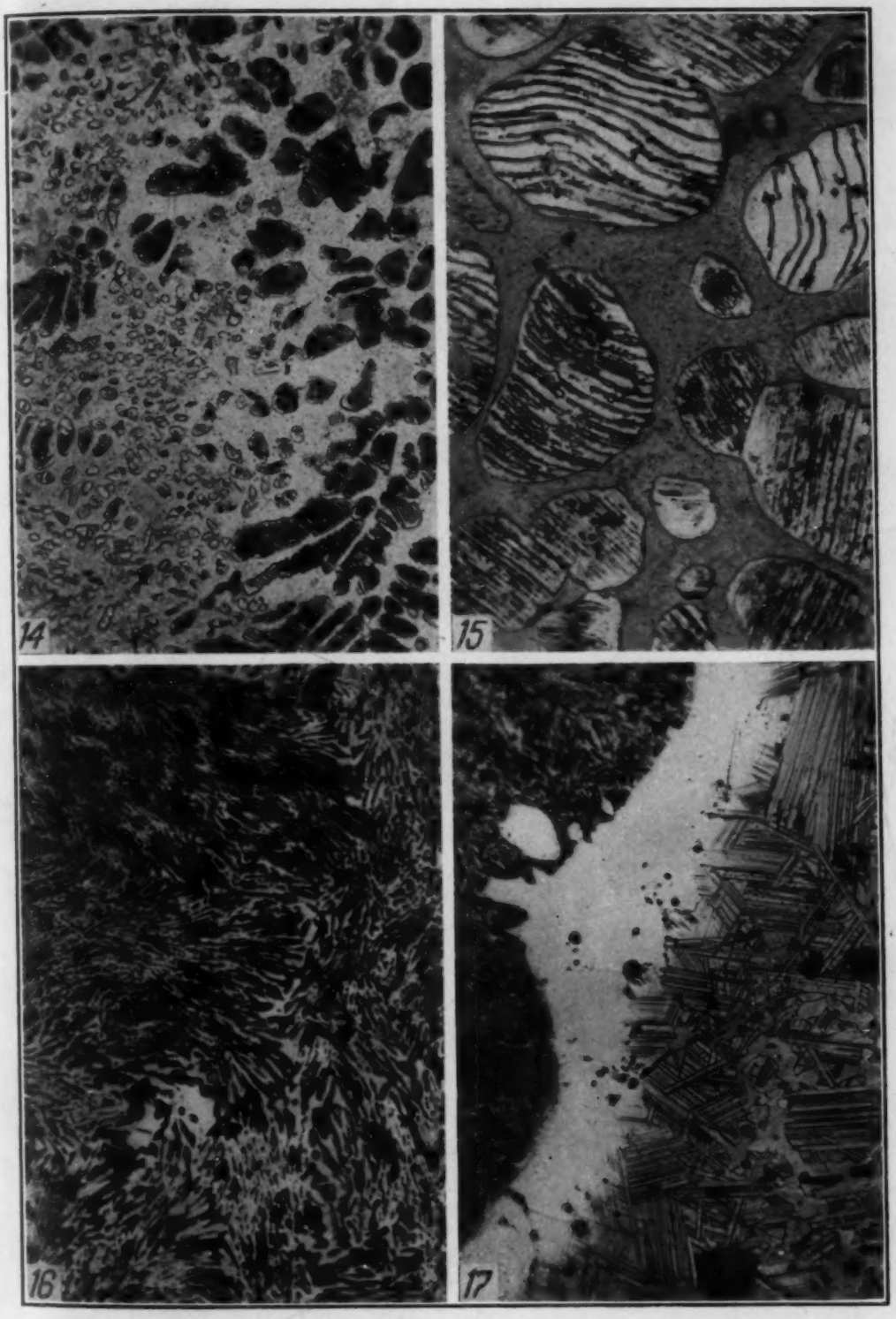


Fig. 14—Tantalum Plus 1.0 Per Cent Carbon. Fused at 2800-2850 Degrees Cent. Primary Ta_2C + Eutectic. × 200.

Fig. 15—Tantalum Plus 1.5 Per Cent Carbon. Fused at 2800-2900 Degrees Cent. Globules of Ta_2C in Tantalum Matrix. × 500.

Fig. 16—TaC-C Eutectic. Formed on Surface of 3.5 Per Cent Carbon Specimen. Heated to 3300 Degrees Cent. Unetched. × 500.

Fig. 17—Structure of Fused Surface of 3.5 Per Cent Carbon Specimen Heated to 3300 Degrees Cent. × 500.

perature shows primary Ta_2C dendrites in a matrix of eutectic (Fig. 14). With increasing carbon content, however, the fine eutectic structure is usually absent and the structure consists of globules of Ta_2C, striated with tantalum, in a tantalum matrix as shown in Fig. 15. Structures consisting almost entirely of Ta_2C show no change from that of the sintered structure after heating above the eutectic temperature.

From the Ta_2C-Ta eutectic point (0.6 per cent carbon) the liquidus curve was observed to rise rapidly with increasing carbon content. The liquidus temperature could be approximated by noting to what extent each specimen of increasing carbon content was fused. However, at 2 per cent carbon and above, the surface of the specimens began to fuse at 3300 degrees Cent. (5970 degrees Fahr.). Observation of the structure of this fused surface showed a eutectic structure of TaC and graphite (Fig. 16). This formation on the surface of a 3.5 per cent carbon specimen may be seen in Fig. 17, wherein the structure of the shell progresses with increasing carbon content from the massive Ta_2C plus mixture of Ta_2C and TaC of the core to a zone of only the mixture of Ta_2C and TaC, thence to solid TaC and finally to the TaC-C eutectic on the surface of the specimen. Analyses of specimens of this eutectic structure showed it to contain approximately 10 per cent carbon.

Due to the rapid absorption of carbon from the crucible as soon as liquid metal was present, the carbon content of the alloys containing the TaC-graphite eutectic was questionable. However, structures to confirm the presence of the eutectic were obtained in various specimens. Fig. 18 shows the structure of primary TaC and eutectic. Fig. 19 shows the typical eutectic structure obtained in most specimens. Excess graphite plus the eutectic may be seen in Fig. 20.

The Ta_2C phase was found to melt at approximately 3400 degrees Cent. (6150 degrees Fahr.). That the melting point of Ta_2C was higher than 3300 degrees Cent. (5970 degrees Fahr.), the TaC-C eutectic temperature, was confirmed by the presence of this eutectic on the unfused core of specimens containing Ta_2C, such as shown in Fig. 17. On heating specimens consisting entirely of the Ta_2C phase of 3.2 per cent carbon to 3400 degrees Cent. (6150 degrees Fahr.), however, the specimens would completely fuse and then consist entirely of the TaC-C eutectic. The completeness of fusion at 3400 degrees Cent. (6150 degrees Fahr.) indicated that this was

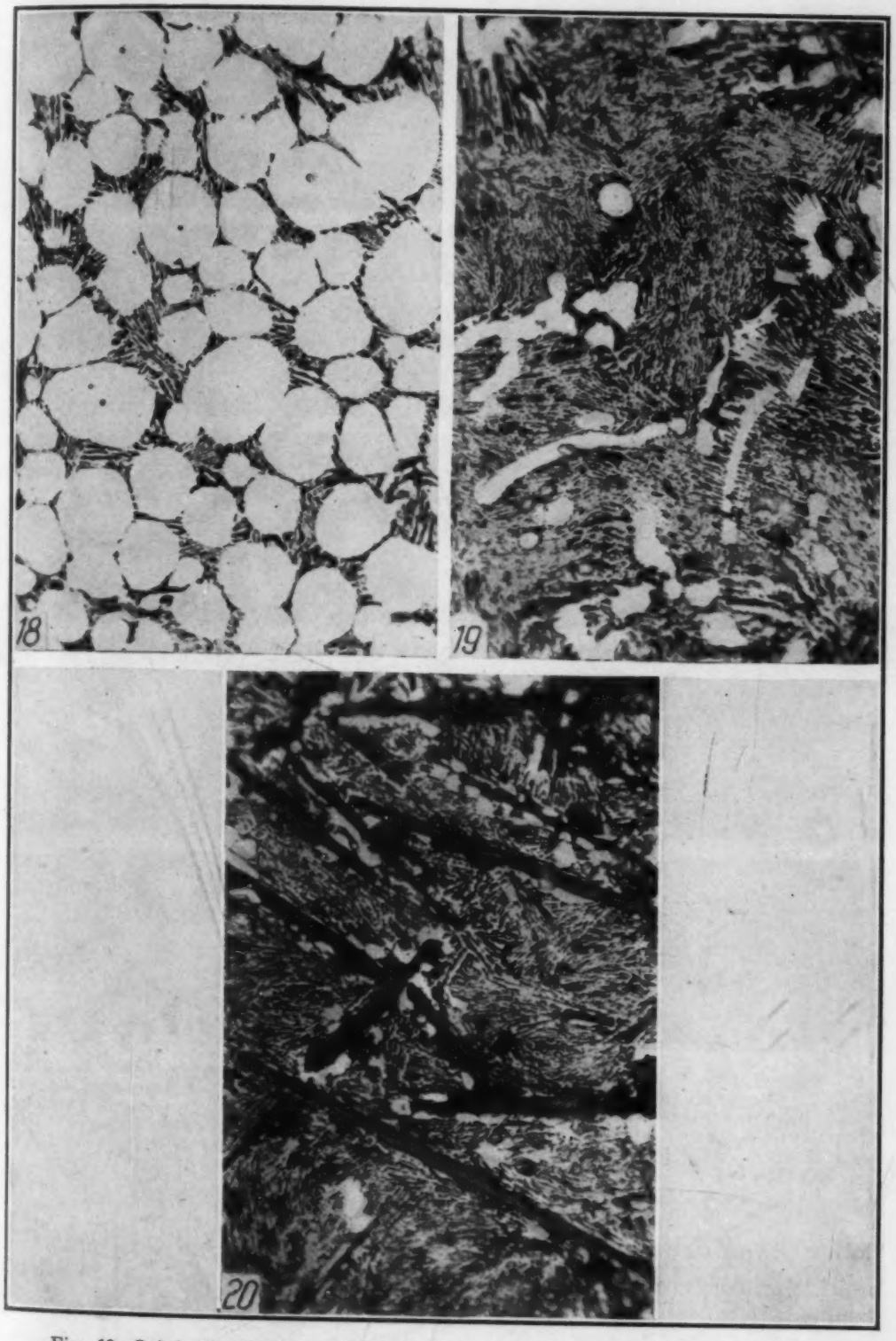


Fig. 18—Originally Tantalum Plus 6.0 Per Cent Carbon. Partially Fused at 3300 Degrees Cent. Micro Shows TaC in Matrix of TaC-C Eutectic. Unetched. × 200.
Fig. 19—Tantalum Plus 10.0 Per Cent Carbon. Fused at 3300-3400 Degrees Cent. TaC-C Eutectic. Unetched. × 200.
Fig. 20—Originally Tantalum Plus 10.0 Per Cent Carbon. Fused at 3300 to 3400 Degrees Cent. TaC-C Eutectic Plus Excess Graphite. Unetched. × 200.

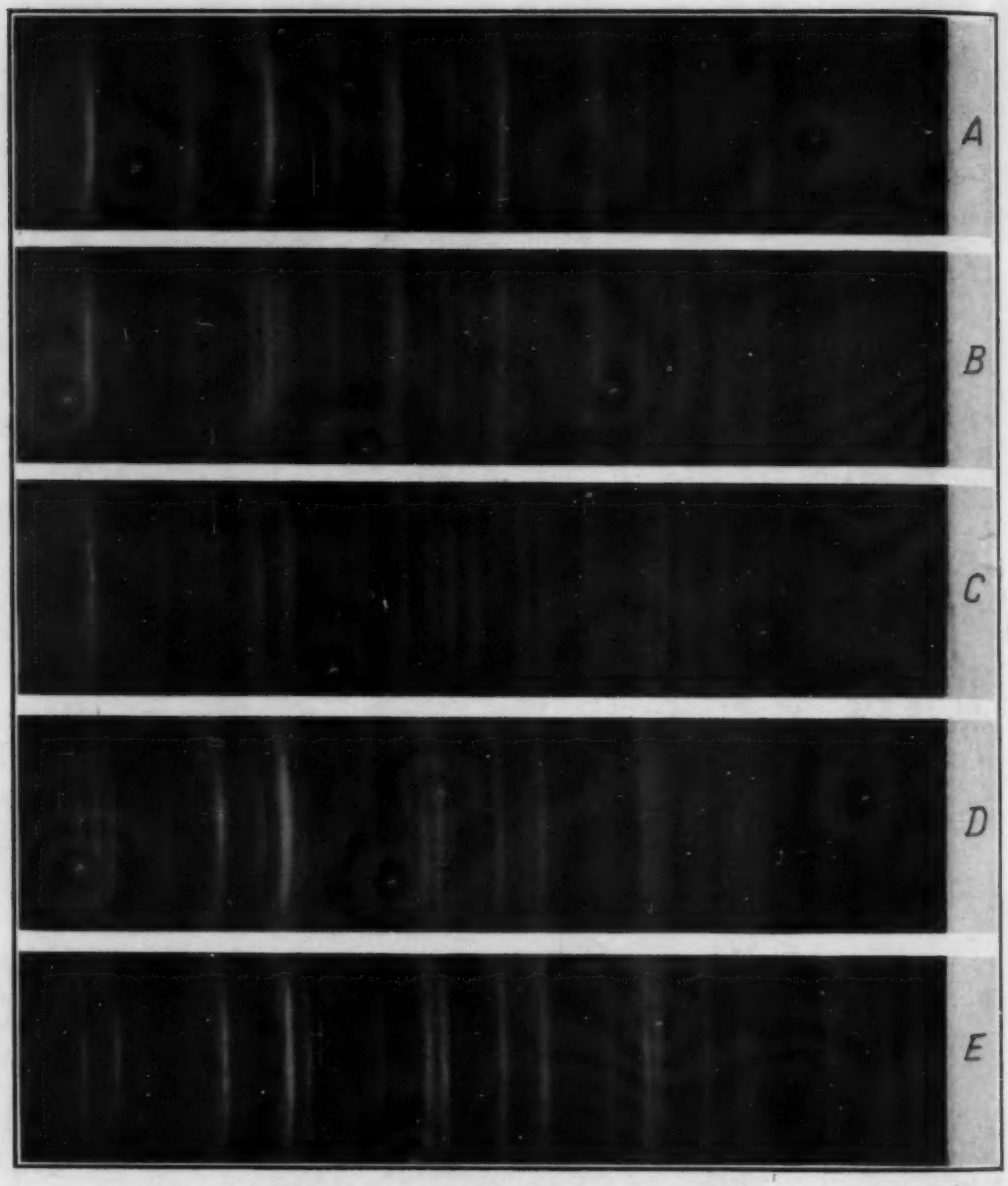


Fig. 21 (A)—Tantalum. Sintered 2700 Degrees Cent.—½ Minute. (B)—Tantalum Plus 1.3 Per Cent Carbon. Fused at 2850 Degrees Cent. (C)—Tantalum Plus 3.2 Per Cent Carbon. Sintered 3000 Degrees Cent.—½ Minute. (D)—Tantalum Plus 4.0 Per Cent Carbon. Sintered 2000 Degrees Cent.—2 Minutes and 3300 Degrees Cent. —½ Minute. (E)—Tantalum Plus 5.0 Per Cent Carbon. Sintered 2700 Degrees Cent. —2 Minutes and 3400 Degrees Cent.—½ Minute.

the approximate fusion temperature of the Ta_2C phase and thus would be the temperature of the peritectic horizontal.

Since the upper temperature limit of the apparatus used was about 3400 degrees Cent. (6150 degrees Fahr.), liquidus temperatures of alloys of more than 2 per cent carbon could not be determined. However, from the foregoing data it seemed very probable that the

liquidus curve kept rising with increasing carbon content up to the melting point of TaC, which was taken as 3800 degrees Cent. (6870 degrees Fahr.) from the work of Friederich and Sittig (1). Beyond the TaC composition, the liquidus falls to 3300 degrees Cent. (5970 degrees Fahr.) at 10 per cent carbon, the TaC-C eutectic point.

X-Ray Data

Diffraction patterns of the various structures observed in the alloys were prepared by the powder method. The metallographic specimens served also for the X-ray samples.

Fig. 21 shows representative photograms produced by alloys of various carbon contents up to 6.2 per cent carbon. Three types of patterns were obtained, namely, those of Ta, Ta_2C, and TaC; thus confirming the metallographic observations of these alloys.

Fig. 21-A is the body-centered cubic pattern of tantalum. Fig. 21-B shows the superimposed reflections of tantalum and of the hexagonal close-packed Ta_2C. Fig. 21-C is the pattern of Ta_2C alone. By the graphic method of Hull and Davey it was identified as being of the hexagonal close-packed lattice and having an axial ratio $(\frac{c}{a})$ of 1.60 to 1.62.

A modification of the Ta_2C phase, as reported by Burgers and Basart (3), was not evident from any pattern secured from the many specimens containing Ta_2C X-rayed during the course of this investigation.

Fig. 21-D shows the superimposed patterns of Ta_2C and TaC and Fig. 21-E that of TaC alone. The TaC lattice is cubic of the NaCl type. Lattice parameter values of 4.445 to 4.56 Angstrom units have been reported in the literature (3).

Polishing and Etching

Alloys up to 3.5 per cent carbon may be polished by the usual methods, but alloys of increased carbon content or which contain an appreciable amount of the hard carbide, TaC, are best polished by the following procedure: A flat surface is ground on the specimen with a carborundum wheel. This surface is further smoothed either on an emery paper or on a copper lap with boron carbide and oil. Final polishing is done on a paper covered wood lap with No. 7

diamond dust and a thin oil. Further improvement of the diamond lapped surface may be obtained by polishing with No. 1 levigated alumina on cloth.

The Ta_2C phase and structures containing this phase are rapidly etched by a mixture of 3 parts of 50 per cent nitric and 1 part of hydrofluoric acid. The TaC phase, however, etches very slowly with this solution, and a mixture of 3 parts of concentrated nitric plus 1 part of hydrofluoric acid gives better results. As the TaC phase progresses with increasing carbon content from white to yellow, it becomes increasingly resistant to the etchant, the yellow TaC being barely attacked at all.

ACKNOWLEDGMENT

The author is grateful to Mr. A. B. Gladding for the preparation of the X-ray diffraction patterns, and to Dr. W. P. Sykes for many helpful discussions and pertinent suggestions.

Bibliography

1. E. Friederich and L. Sittig, "Herstellung und Eigenschaften von Carbiden", *Z. anorg. allg. Chem.*, Vol. 144, 1925, p. 174-181.
2. C. Agte and H. Alterthum, *Z. techn. Physik*, Vol. 11, 1930, p. 185.
3. W. Burgers and J. Basart, "Über die Bildung hochschmelzender Metallcarbide beim Glühen eines Kohlenfadens im Dampf einer flüchtigen Halogenverbindung des Metalles," *Z. anorg. allg. Chem.*, Vol. 216, 1934, p. 209.
4. W. P. Sykes, "A Study of the Tungsten-Carbon System," TRANSACTIONS, American Society for Steel Treating, Vol. 18, 1930, p. 968.

DISCUSSION

Written Discussion: By F. C. Kelley, Research Laboratory, General Electric Co., Schenectady, N. Y.

I was interested in reading Mr. Ellinger's paper because he describes his work as if it were just one of those things accomplished in the regular routine of everyday duties. Those who are familiar with the properties of tantalum and its activity towards all gases, water vapor, etc., might well wonder how it could be done so easily. He has done a very fine piece of work which checks with all of the observations I have made on the tantalum-carbon system.

There is one very interesting point which the author brings out in his discussion of the diffused layers on the piece of tantalum rod heated in the Ajax vacuum furnace at 2300 to 2400 degrees Cent. (4170 to 4350 degrees Fahr.) for one-half hour, namely, that each of the two distinct shells is composed of two layers.

Frederick N. Rhines in a paper, "Diffusion Coatings on Metals," presented before this society at the fall meeting of 1940, brought out very clearly that

the layers formed in binary systems by diffusion at a substantially constant temperature and pressure correspond in kind and in order of their occurrence to the single-phase regions in the phase diagram at the temperature and pressure of diffusion and no two-phase layers appear. He also brought out the point that layers corresponding to both the one- and two-phase regions appear in ternary systems.

When we consider the work of Mr. Ellinger as shown in photomicrograph Fig. 2, it would seem that here we have a binary system which exhibits very broad two-phase layers equal in size or larger than the single-phase layers. Perhaps this is an exception to the rule laid down by Dr. Rhines.

In the last paragraph of the author's discussion of materials and apparatus he attributes the drop in vacuum, 1-3 millimeters of Hg, to the vapor pressure of carbon. When we consider the large quantities of gas absorbed and adsorbed by carbon it would seem that the release of this gas alone would be sufficient to account for the drop in pressure. Graphite continues to give off gas with every increase in temperature. When the heat is turned off and it is cooling down it is capable of performing the duty of a high speed molecular pump.

I wish to congratulate the author on his fine piece of work and his contribution to scientific knowledge by adding another binary system to the list.

Oral Discussion

F. N. RHINES:[1] There are two ways in which two-phase layers might have been produced by diffusion in these alloys: First, by the decomposition of a one-phase layer during cooling, and second, by the presence of a third component. The former is the more probable explanation where there appears to be a gradual transition from a one-phase to a two-phase layer. Such a structure results from a concentration gradient in the original one-phase layer; upon cooling, decomposition occurs where the saturation limit has been exceeded. Two-phase layers in ternary systems are usually bounded by more definite limits than those shown in the photomicrographs of this paper.

It is my guess, therefore, that the case shown here is due rather to the decomposition of a one-phase layer than to the presence of impurities.

Upon another subject, may I remark that the curved striations of tantalum across the Ta_2C in Fig. 15 suggest eutectoid decomposition rather than simple precipitation from solid solution. Simple precipitation in the solid state usually results in a more geometrically perfect structure. Perhaps there is a reaction $Ta_2C \rightarrow Ta + TaC$ at low temperatures.

HENRY S. RAWDON:[2] I would like to ask about possible variations in hardness.

MR. ELLINGER: I can answer that by saying when you come down to the pure alloys, they are always porous and very brittle and no hardness measurements could be made on them very well.

DR. RAWDON: That is just the point I want to make. We usually consider that hardness cannot be made except on rather large, gross scale, but with the

[1]Assistant professor, department of metallurgy, Carnegie Institute of Technology, Pittsburgh.

[2]Chief, division of metallurgy, National Bureau of Standards, Washington, D. C.

instrument recently developed at the Bureau of Standards, which is now on the market as the Tukon hardness tester, we can measure the hardness of very small particles of an alloy constituent. It would be very interesting in a case like this to know what the hardness of those two carbides is, and of the plates of the metal in between. That could easily be done with an instrument of that kind.

MR. ELLINGER: No attempt was made to make any hardness measurements.

Author's Reply

I wish to thank Dr. Rhines for explaining those carburized shells so well. However, a third element may enter into the system which could be nitrogen. It has been reported in the literature that certain workers have thought there was nitrogen present, but otherwise, during the course of this work, no three-phases were observed in the alloys, that is, in the alloys which were conducted in the vacuum.

Burgers and Basart in the German paper have reported a modification of the Ta_2C phase from the high temperature down to the low temperature. During this investigation, only one diffraction pattern of Ta_2C was obtained. In the many specimens X-rayed, no two modifications showed up. However, one of them may be a high temperature modification which could not be retained at room temperature. If such is the case, there might very well be a eutectoid decomposition at some lower temperatures, resulting in these tantalum striations which Dr. Rhines spoke about.

However, if there is a modification in the Ta_2C phase, I feel that it occurs some place above 2500 degrees Cent. (4530 degrees Fahr.).

Since the presentation of this paper, hardness measurements of TaC and Ta_2C have been made with the Knoop Micro-Hardness Tester by E. R. Parker of the General Electric Company.

Due to the fine porosity of the specimens it was difficult to obtain consistent measurements; however, the micro-hardness of TaC was found to be around 2000 and that of Ta_2C around 1000.

THE METHOD OF THIN FILMS FOR THE STUDY OF INTERMETALLIC DIFFUSION AND CHEMICAL REACTIONS AT METALLIC SURFACES

By Howard S. Coleman and Henry L. Yeagley

Abstract

A simple method for observing intermetallic diffusion rates has been devised. This method involves the deposition from the vapor phase first of one metal then another on top of it. A microscope slide is used as a film base. The diffusion rates are then observed at various temperatures. This is done by noting the change in reflectivity at one surface as the metals diffuse.

The apparatus consists essentially of an automobile headlight source maintained at constant voltage, the bimetallic reflecting film, and a photronic cell connected to a recording microammeter. The recorder draws a record of the change in reflectivity automatically as the diffusion progresses.

This new method makes possible the study of diffusion rates of metals through metals and, in some cases, gases through metals at temperatures as low as 50 degrees Cent. and in lengths of time as little as 5 minutes, depending upon the metals involved, the thickness of the metals, and the temperatures.

The activation energies determined by the method of thin films are in agreement with values obtained by others. For example, the activation energy for the diffusion of gold into lead has been found to be —13700 ±300 calories.

Introduction

THE rate at which one solid metal diffuses into another is one of great practical and theoretical importance. The methods that have been used by previous investigators are very adequately described by Barrier,[1] and, therefore, will not be reviewed here.

It is the purpose of this paper to present a new and greatly

[1] R. M. Barrer, "Diffusion In and Through Solids," May 1941, pages 306-310.

A dissertation by Howard S. Coleman submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy at The Pennsylvania State College, May 1942.

A paper presented before the Twenty-fourth Annual Convention of the Society held in Cleveland, October 12 to 16, 1942. The authors, Howard S. Coleman and Henry L. Yeagley, are associated with the department of physics, The Pennsylvania State College, State College, Pa. Manuscript received May 7, 1942.

simplified method of observing diffusion rates. This new method eliminates a number of objectionable features that exist in other methods such as:

1. The difficulty of obtaining intimate contact between the two diffusing metals.
2. The very long time required to observe a measurable amount of diffusion.
3. The necessity of having to observe diffusion at high temperatures.

The quantitative methods used in the study of intermetallic diffusion involve measurement of the factors found in Fick's[2] law. This law in equation form is:

$$dm = D\ A\ \frac{dc}{dx}\ dt \qquad (1)$$

This indicates that the amount of metallic solute that diffuses across an area, A, at a point where the concentration gradient in the solution of metals is $\frac{dc}{dx}$, in the time, dt, is equal to dm. The constant, D, depends upon the temperature, the units used, and the concentration of the solute.

Another equation is obtained from (1) by assuming that D does not depend upon the concentration (and hence x). This assumption is valid for very dilute solutions.

$$\frac{dc}{dt} = D\ \frac{d^2c}{dx^2} \qquad (2)$$

The integral of equation (2) will be discussed later in conjunction with the calculations. As far as this report is concerned, however, the most important contribution made by Fick's law is the listing of the factors that influence the rate of diffusion. The quantities that are usually measured in diffusion studies are therefore:

a. $\frac{dm}{dt}$

b. $\frac{dc}{dx}$

c. A

d. The temperature

e. c

[2] A. Fick, Pogg. Ann., Vol. 94, 1855, p. 59.

Experimentally the diffusion constant, D, is found to be a function of the absolute temperature. D is of physical significance because it is a measure of the "activation energy". Mehl[3] describes this "activation energy" as being related to the amount of energy required to "loosen up" the lattice of the solvent and permit diffusion. For the cases that have been studied

$$D = Be^{-\frac{b}{T}} \qquad (3)$$

B = constant
T = absolute temperature

The quantity, b, usually constant, may be rewritten as $\frac{Q}{R}$ where R is the gas constant. Since R T assumes the physical dimensions of energy, Q must also represent energy. Q is defined to be the "activation energy".

The Method of Thin Films

The Method of Thin Films involves the deposition from the vapor phase on a film base first of one metal and then of another on top of it. The film base bearing the two metallic layers is then heated to the desired temperature, and the progress of the diffusion is observed by measuring the change of reflectivity as one metal diffuses into the other.

The Apparatus and Procedure—The discussion of the apparatus and procedure is divided into the following parts:

1. The vaporization chamber
2. The diffusion apparatus
3. The data
4. The calculation and discussion of results

The Vaporization Chamber—The vaporizing chamber is part of the vacuum system pictured in Figs. 1 and 2. The vaporization is made from two 120-mil tungsten rod filaments as shown in Figs. 3 and 4. The filaments have cavities ground in them about 1 centimeter long, 2 millimeters wide, and 1 millimeter deep. The metals to be evaporated are placed in these cavities and a current of up to 420 amperes at 10 volts passed through the filaments. The current used

[3]Mehl, *Transactions*, American Institute of Mining and Metallurgical Engineers, Vol 122, 1936, p. 11.

Fig. 1—The Vacuum System.

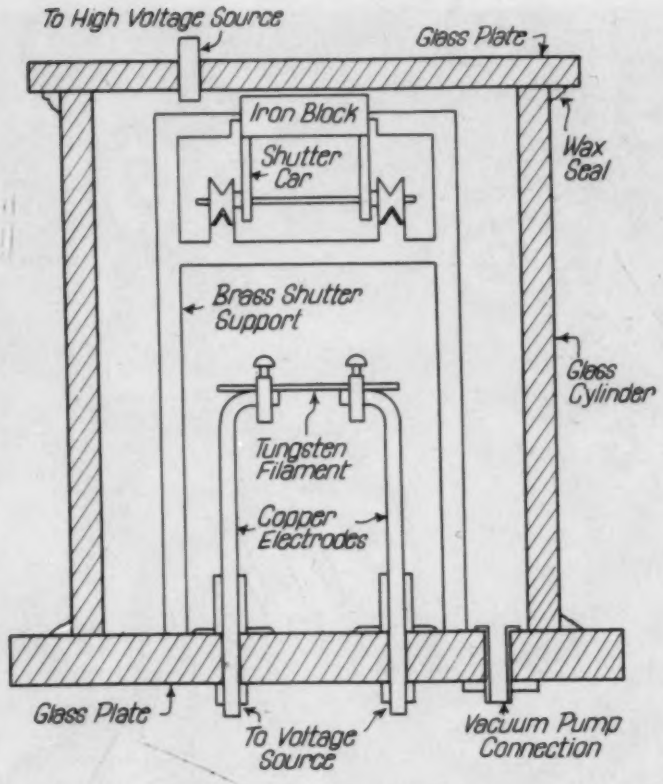


Fig. 2—The Vaporizing Chamber.

depends upon the boiling point of the particular metal to be vaporized.

Three microscope slides are suspended above the filaments from a shutter mechanism (Fig. 4 and Fig. 5). This shutter is arranged with three slots, one for each slide in such a way as to be able to close off either end slide but not the middle one. The shutter is set in one position and one metal vaporized. The shutter is then shifted to the other position and the second metal vaporized. By this method the center slide is coated by each metal and each end slide with only one metal. The purpose of the two end slides is to furnish a means of determining the thickness of each film of the two metals. This is possible since the surface density is very nearly constant over the area occupied by slides. The uniformity of the surface density was checked by placing a number of small sections of slides in the region normally used and a metallic film deposited on them. The surface density was then determined for each section. The maximum percentage difference of the surface density of the section nearest the filament, 25 centimeter, and the surface density of the section farthest from the filament, 28 centimeter, was 2. The end slides with the metal films on them are weighed. Then the films are removed, and

Fig. 3—The Vaporizing Chamber.

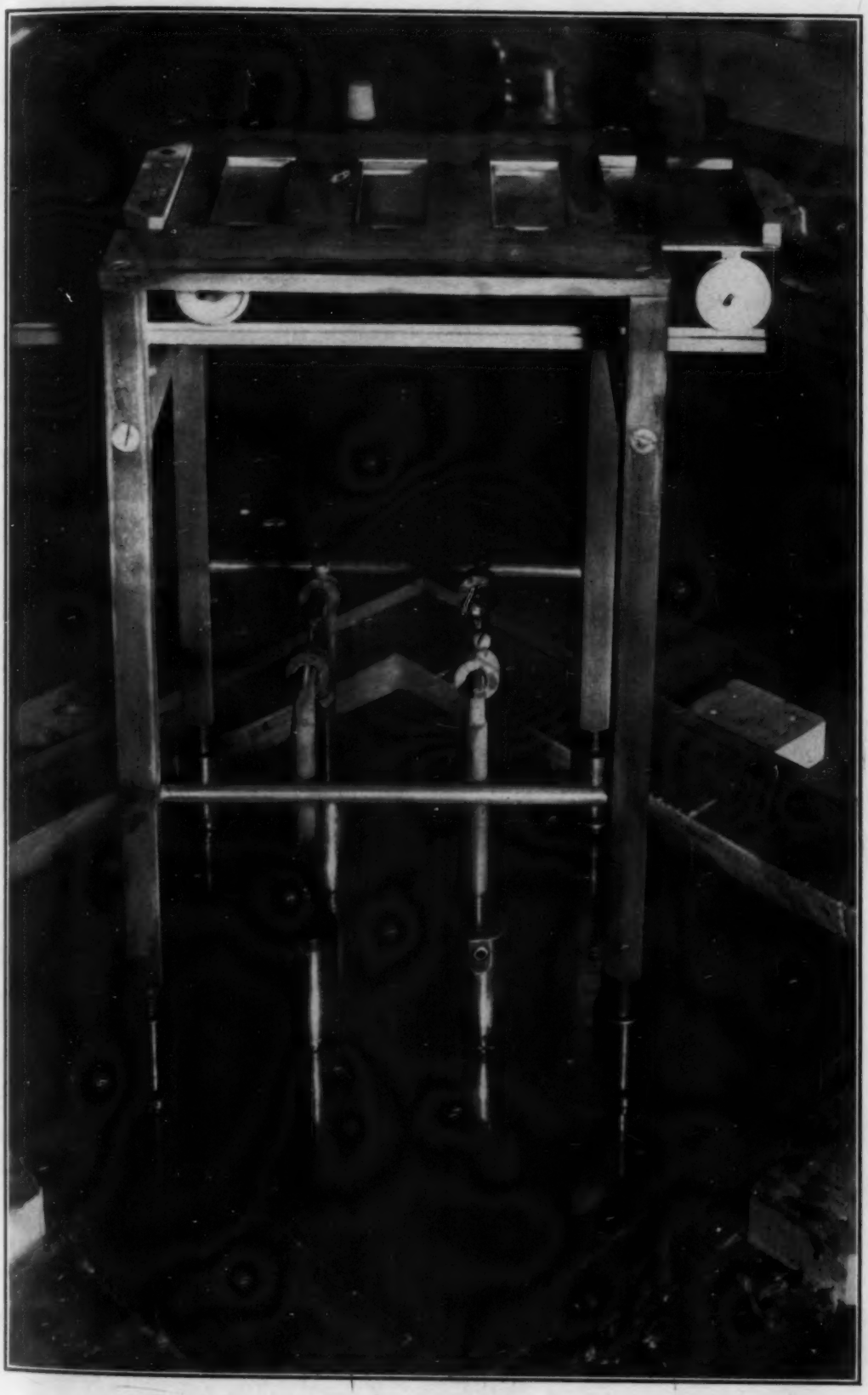


Fig. 4—The Shutter.

the slides weighed. The difference between two such weighings gives the weight of the film. Since the areas and densities of the deposits are known, it is possible to calculate the thickness of each film.

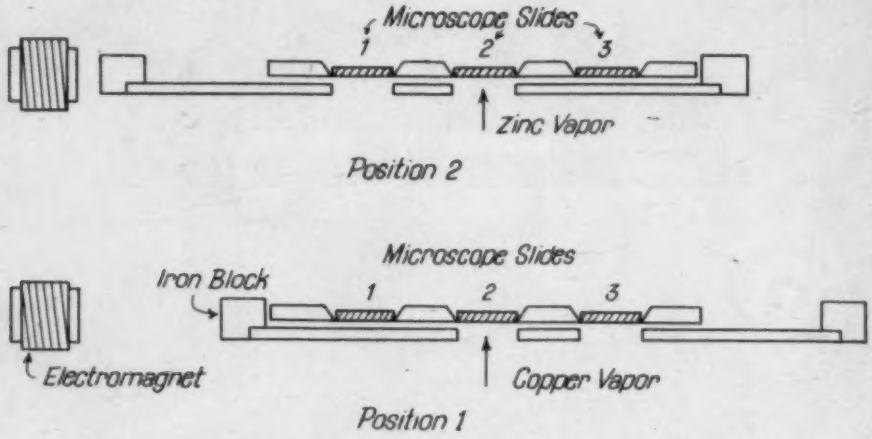


Fig. 5—The Shutter.

The Diffusion Apparatus—The diffusion apparatus is really a reflectometer. This reflectometer (Figs. 6 and 7) consists essentially of an automobile headlight source, the bi-metallic coated slide as a reflector, and a photronic cell as receiver. The photocell is connected to a self-recording microammeter which draws a record of the change in reflectivity as the diffusion progresses. (In Figs. 6 and 7 the apparatus is pictured as it was when used for the study of the rate of oxidation of copper, which is not discussed in detail here.)

The temperature is controlled by an ordinary flatiron with a built-in thermostat, and the temperature is held constant within 1 degree Cent. The iron itself sits on a ¼-inch brass plate in which is milled a slot to hold the microscope slide which has both metals deposited on it. The actual surface used in the reflection is only about 1 centimeter square. No appreciable temperature gradient was found over this area. A chromel-alumel thermocouple placed about 1 millimeter from the metal films was used to measure the temperature.

The Data

The data are presented in graphical form. All the weights and thicknesses for a particular diffusion are listed on the graph itself.

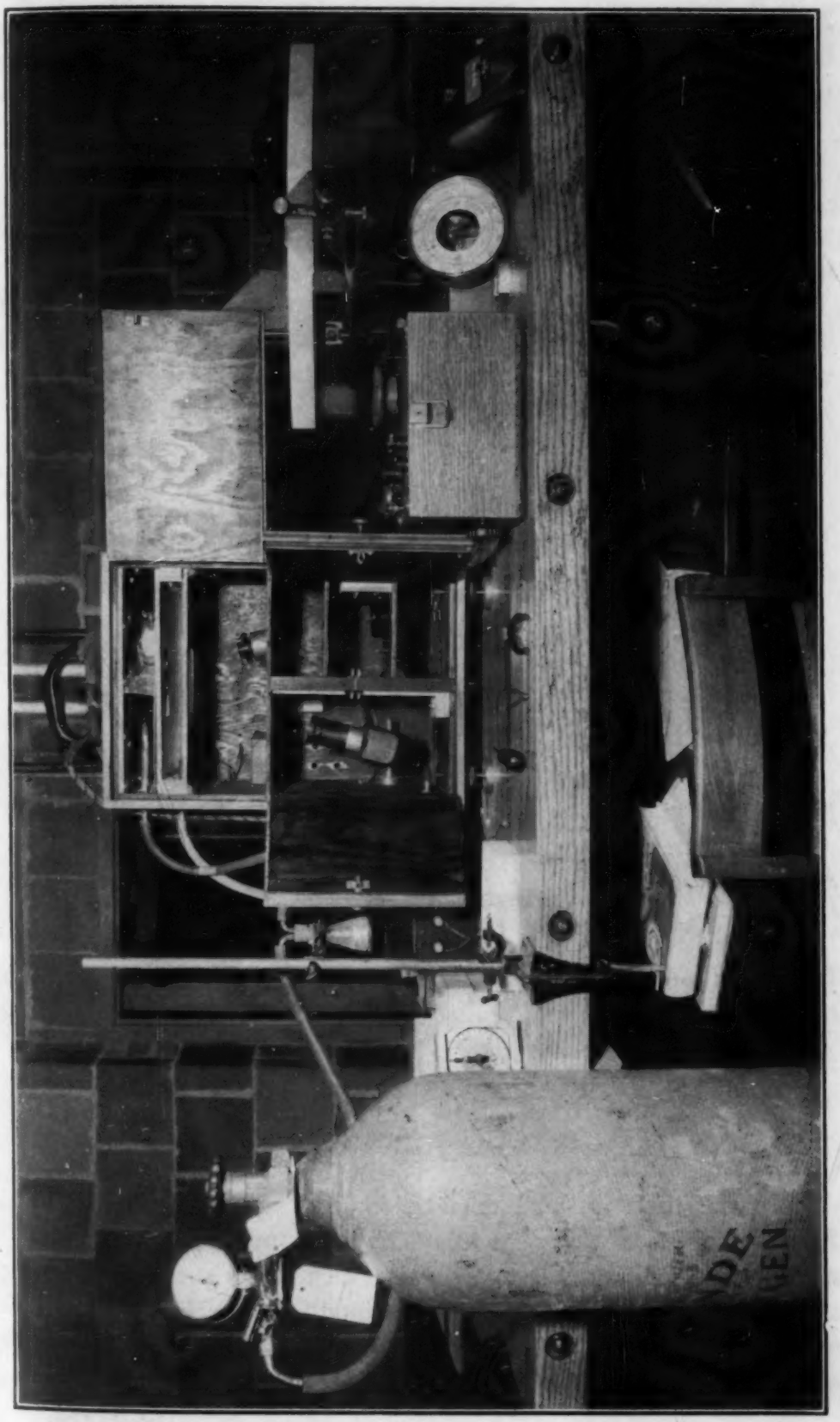


Fig. 6—The Diffusion Apparatus.

Although the diffusions of a number of bi-metallic systems have been observed, only two sample systems are given here to illustrate the application of the method.

Calculations and Discussion of Results

The Activation Energy—The usual method of determining Q, the so-called activation energy, for the diffusion is as follows:

The relation between D and the atomic per cent of the solute is obtained. D is then extrapolated to zero atomic per cent. The value D_o thus obtained is then plotted in the following fashion:

$$\text{Log } D_o \text{ vs. } \frac{1}{T}$$

The slope of the resulting straight line is proportional to Q since the relation between D_o and T is:

$$D_o = Be^{-\frac{Q}{RT}} \qquad (4)$$

where B = constant depending on units only
R = gas constant
Q = activation energy

By taking the logarithm of both sides of (4) we get:

$$\text{Log}_e D_o = \text{Log}_e B - \frac{Q}{RT} \qquad (5)$$

In practice it is customary to use common logarithms in which case, since $\text{Log}_e N = 2.3 \text{ Log}_{10} N$, the slope, b, of the line $\text{Log}_{10} D_o$ vs. $\frac{1}{T}$ is $-\frac{Q}{2.3R}$ from which

$$Q = 2.3 \text{ Rb.}$$

In the present stage of development of the Method of Thin Films, it appears advisable to modify this method of obtaining Q although there is no reason why the customary procedure outlined above could not be followed. The modification eliminates the determination of D_o which is time consuming and unnecessary as far as the determination of Q is concerned.

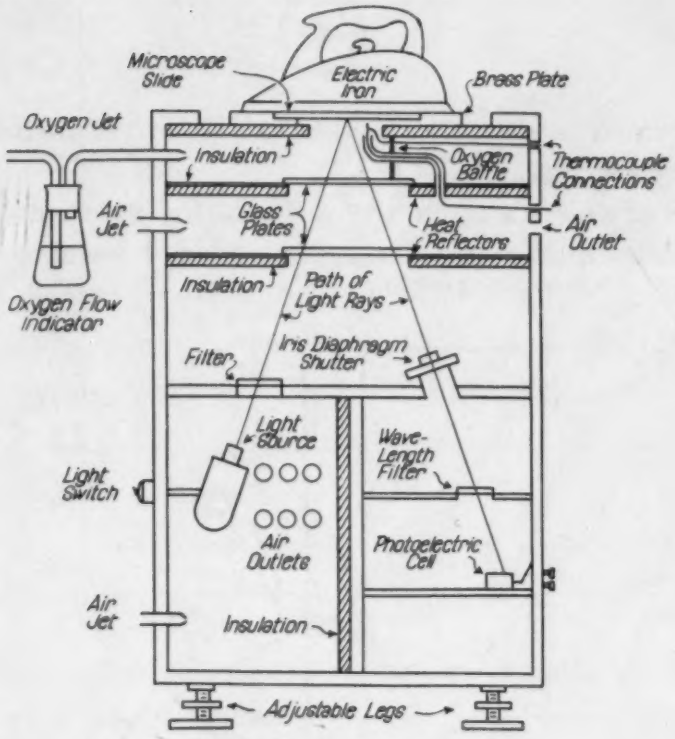


Fig. 7—The Diffusion Apparatus.

Let us define a quantity t_∞ as follows

Let t_∞ be the time at which $\frac{d\,R}{d\,t} = a$; and let $a = 0.00291$, i.e., the time at which the slope of the reaction rate curve is tan 10′. Any arbitrary angle could be used but by choosing "a" in this fashion, we are very close to having a homogeneous alloy which fixes desirable boundary conditions.

Experimentally, t_∞ depends upon the conditions of the experiment in the following manner:

It is directly proportional to the thickness, x, and is also proportional to the amount of metal, m, that must pass the interface in the positive direction of x in order to bring about homogeneity of the alloy. That is:

$$t_\infty = h\,x\,m$$

where h is a proportionality constant depending upon the temperature.

It is convenient to define a quantity $K = \frac{1}{h}$ to express the results in the form of a rate, i.e.:

$$K = \frac{x\,m}{t_\infty} \qquad (6)$$

K is then treated in the same manner as D_o in the method outlined above for the calculation of Q.

As an example, consider the diffusion of gold into lead using Fig. 8, and substituting gold for copper and lead for zinc. At $t = t_\infty$ the lead and gold films have formed a homogeneous alloy. The

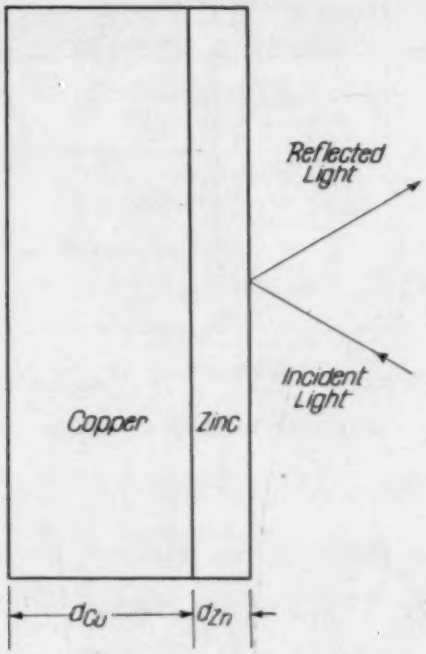


Fig. 8—The Bi-Metallic Film.

amount of gold actually having passed the interface is then the amount of gold occupying the fraction of the total volume of the alloy which was initially occupied by lead.

$$m = \frac{W_{Au}\ d_{Pb}}{(d_{Au} + d_{Pb})} \qquad (7)$$

the fraction of the weight of gold between $x = O$ and $x = d_{Pb}$.

$$x = d_{Pb} \qquad (8)$$

the initial thickness of the lead.

$\therefore$ from (6), (7), and (8)

$$K = \frac{d_{Pb}^2\ W_{Au}}{(d_{Au} + d_{Pb})\ t_\infty} \qquad (9)$$

experimentally (as indicated by Fig. 10):

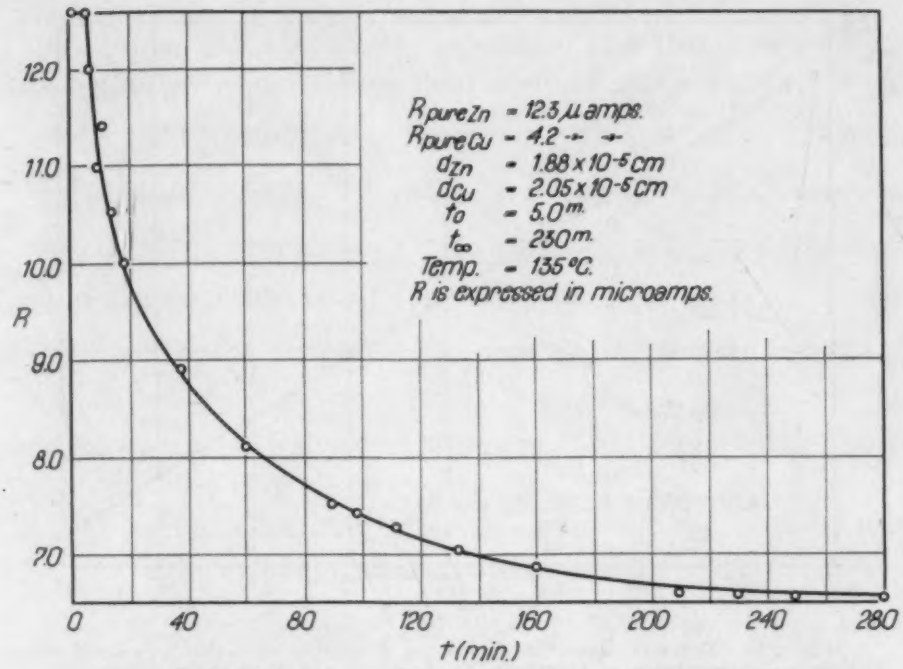


Fig. 9—Reflectivity Curve for the Diffusion of Copper Into Zinc.

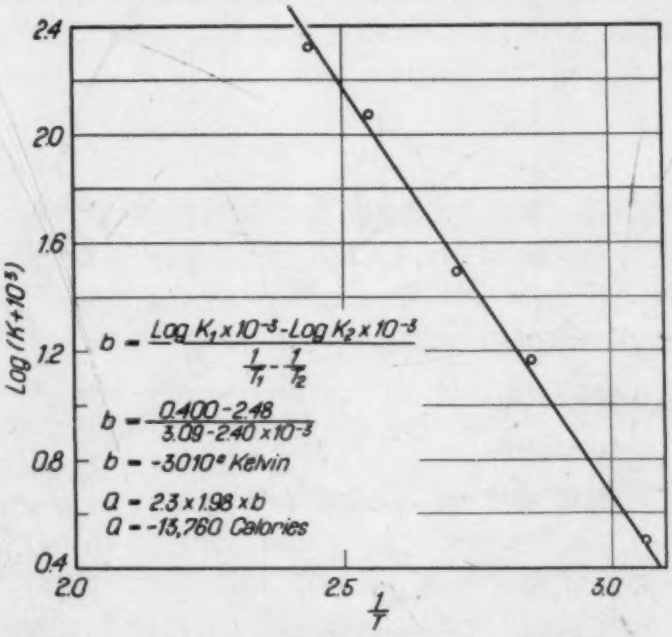

Log (K×10³)
$b = \frac{\text{Log } K_1 \times 10^{-3} - \text{Log } K_2 \times 10^{-3}}{\frac{1}{T_1} - \frac{1}{T_2}}$
$b = \frac{0.400 - 2.48}{3.09 - 2.40 \times 10^{-3}}$
b = -3010° Kelvin
Q = 2.3 x 1.98 x b
Q = -13,760 Calories
$\frac{1}{T}$

Fig. 10—Activation Energy Curve for the Diffusion of Gold Into Lead.

$$K = Ee^{-\frac{Q}{RT}}$$

$$\therefore \text{Log}_{10} K = \text{Log}_{10} E - \frac{Q}{2.3\ R\ T} \qquad (10)$$

A plot of Log_{10} K vs. $\frac{1}{T}$ is treated in the same fashion

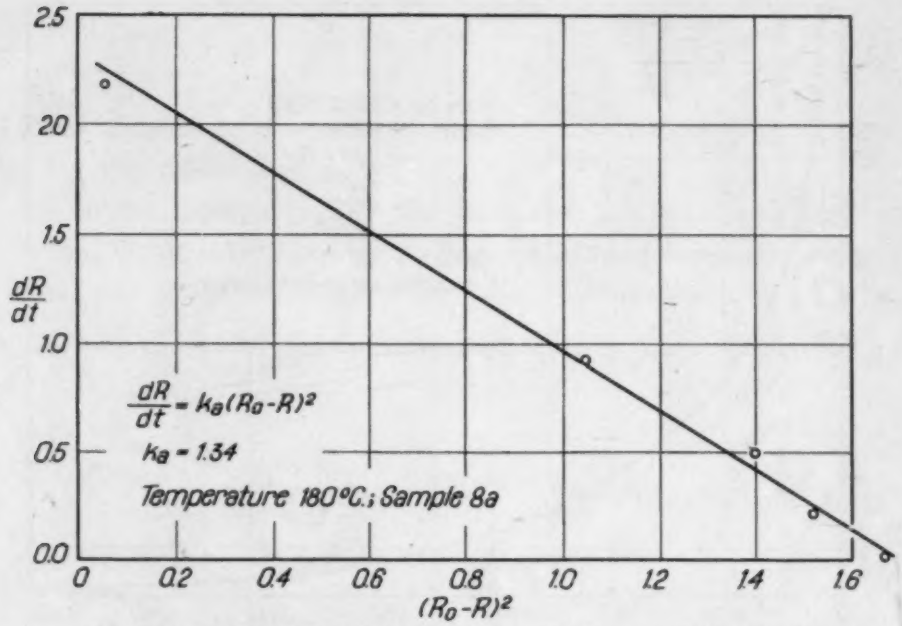


Fig. 11— Reaction Rate Graph for the Diffusion of Gold Into Lead at Different Temperatures at Constant Thickness of the Bi-Metallic Film.

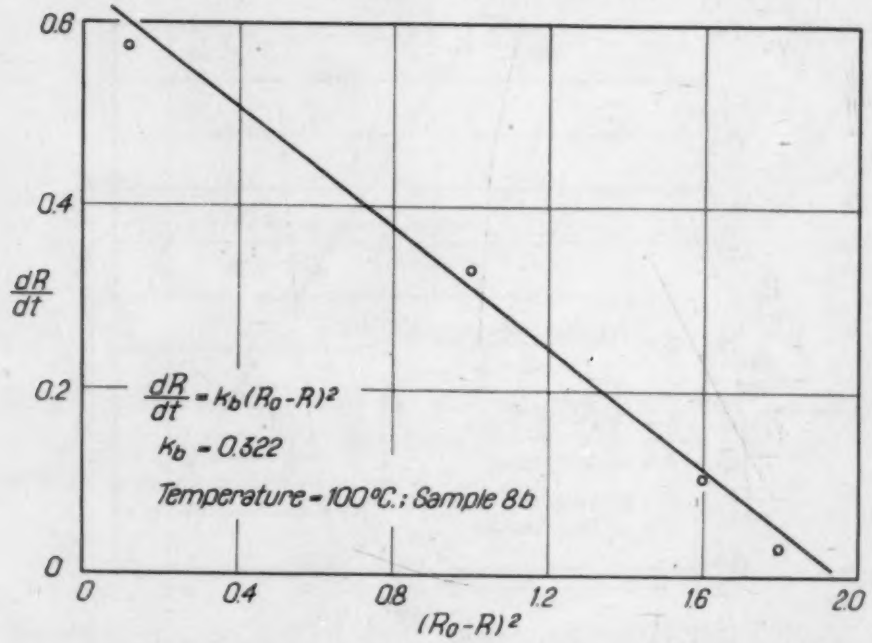


Fig. 12—Reaction Rate Graph for the Diffusion of Gold Into Lead at Different Temperatures at Constant Thickness of the Bi-Metallic Film.

as D_o. Fig. 10 shows the results of such a procedure for the diffusion of gold into lead.

According to a survey of the progress of intermetallic diffusion made by Mehl,[3] the value of Q for the diffusion of gold into lead is —13,600 calories. The slope of the line in Fig. 10 by the Method

of Thin Films gives —13,700 calories. The percentage difference between the two values is less than the probable error of the experiments.

Calculation of the Equations for Reaction Rate Curves

The equations of reaction rate curves for the diffusion of gold into lead are found to be of the following form:

$$\frac{dR}{dt} = k\,(R_o - R)^n$$

where R_o = the reflectivity of the pure metal from which the reflection is made at $t = t_o$.

R = the reflectivity after the diffusion has been in progress for a time, t, measured from t_o.

Figs. 11 and 12 indicate that $n = 2$. A graph of Log k vs. $-\frac{1}{T}$ is a straight line if the thicknesses are the same in all cases. The calculation of Q from k gives —14,000 calories, which is substantially the same result as the other method outlined in this report.

Conclusions

The conclusions are in the following brief statements:

1. Fick's[2] law may be applied to thin films.
2. The values of the energies of activation by the Method of Thin Films are in agreement with the values obtained by other methods.
3. The process of putting the two metals in contact by vaporization in a vacuum gives very intimate contact.
4. The time required to observe a diffusion is tremendously shortened by the Method of Thin Films.
5. Diffusion of metals may be observed at much lower temperatures by the Method of Thin Films than by the other methods.
6. The reflection technique is a simple method of observing diffusion.

Comments on the Method of Thin Films

1. It is evident that the reflection technique is most satisfactory for metallic systems in which the components are of

different color. Plans have been made to use the Method of Thin Films to study bi-metallic systems whose components are the same color by X-rays and electron diffraction.

2. There are other uses for the Method of Thin Films and the reflection technique: they may be used to study reactions occurring at metallic surfaces in which a chemical compound is formed that has a different reflectivity than the metal itself. In this connection, the rate of oxidation of copper was studied. Although a great deal of data have been obtained for the copper-oxygen system, they will be presented in a separate report.
3. It is probable that the use of thin films will initiate changes in the mathematical analysis used in the study of intermetallic diffusion.
4. The purity of the metals is known to affect the rates of diffusion. The zinc and copper used in these experiments were of exceptionally high grade and were supplied by the New Jersey Zinc Company and the American Smelting and Refining Company. The gold used was in the form of pellets made from noncohesive, 24 carat, gold foil obtained from S. S. White, Dental Manufacturing Company. The lead was "Baker's Analyzed" c. p. grade.
5. In certain bi-metallic systems either or both of the components may not be in their normal crystalline structures. That is to say, after the metal vapor is condensed, the normal crystal structure will not be resumed unless the surface on which it is condensed is at a sufficiently high temperature. This fact should be considered when comparisons are made between diffusion coefficients measured at greatly different temperatures.
6. The precision of the present measurements in the determination of the activation energy is about 2 per cent. A probable value of Q from the diffusion of gold into lead is $-13{,}500 \pm 300$ calories.
7. Diffusion of certain gases through certain metals can be studied by observing the change in reflectivity as the gas completely penetrates the film.
8. In certain bi-metallic systems the reflectivity curves pass through minima. These minima occur in the zinc-copper system when the ratio of the thickness of copper to the thick-

ness of zinc is greater than 3 approximately. It is likely that the minima occur at the time when the composition (of the brass formed in the diffusion) is changing from one solid phase to another.

9. A mathematical analysis for the calculation of the diffusion coefficient, D, is being developed and will be presented later.

Acknowledgments

The authors wish to express their thanks to Dr. R. F. Mehl and Dr. F. N. Rhines of the Metals Research Laboratory of the Carnegie Institute of Technology for their interest and help.

They are also greatly indebted to Dr. W. R. Ham, Head of the Department of The Pennsylvania State College, who directed the research of one of us in connection with a dissertation submitted to the Graduate School of The Pennsylvania State College.

DISCUSSION

Written Discussion: By W. R. Ham, head of physics department, The Pennsylvania State College, State College, Pa.

It will occur to anyone that the emissivity of surfaces could be used at the higher temperatures as well as reflectivity. Dr. Wayne Webb and I tried this four years ago and the preliminary work was quite promising.

Obviously a continuous X-ray analysis of one surface could be undertaken, and methods are now available as developed, for example in Dr. W. P. Davey's laboratory, that would be satisfactory.

So the examination of a surface to determine when and to what extent another element has reached this surface is not too difficult.

The most appealing point to me of the intermetallic diffusion process described by the authors is the short time required, and the low temperatures at which it is feasible to work.

A helpful contribution from metallurgists would be suggestions of needed diffusion data that could be obtained by such apparatus.

Written Discussion: By F. C. Whitmore, Dean, The Pennsylvania State College, State College, Pa.

From the chemical point of view, The Method of Thin Films makes it possible to check independently a number of important thermodynamic quantities involved in the reactions of metals with gases. These include such quantities as reaction velocity constants, entropy changes, specific heats, dissociation pressures, and heats of formation as functions of temperature. In most gas-metal systems, no such studies have been possible because of the technical difficulties involved. This new method has merit in that it eliminates many of these difficulties and makes possible techniques altogether simpler than those of the classical methods.

The new method will undoubtedly be of value in obtaining data for constitution diagrams of certain alloys. The study of diffusion is not limited to pure metals but can be applied to certain alloys such as brasses.

The time may even come when this technique will offer an opportunity for new studies on the action of various organic compounds on metals.

Written Discussion: By A. F. Turner, Scientific Bureau, Bausch & Lomb Optical Co., Rochester, N. Y.

This method for determining intermetallic diffusion by employing thin evaporated films of the metals in question promises to become of value in studying the structure of the films themselves. It is known that by controlling deposition conditions evaporated or sputtered films can be obtained which differ considerably in physical and optical properties from the parent massive metals. It might be expected that a change in activation energy would be observed if one metal of a diffusing pair were made intentionally with, for example, a density lower than normal. Furthermore, as the authors point out, the diffusion process is affected by impurities so that it would seem possible to study their effect on films by this method. If such aspects of film structure could be examined by the authors' convenient reflection procedure, a welcome additional means of thin film investigation would be provided.

Oral Discussion

HAROLD E. TRENT:[4] I was wondering whether this method could be applied to the explanation for the deterioration of platinum thermocouples and to what extent.

MR. COLEMAN: The question has come up as to whether or not one could use such a technique to study the deterioration of platinum thermocouples and, of course, one would have to use the emissivity method as proposed by Dr. Ham just a moment ago at the highest temperatures, but if one were studying the emissivity as a function of temperature and found some peculiarity in the functional relation, indicating some peculiar change, order, disorder, or something like that, if such a thing were possible then emissivity methods could be used, but it is questionable. It would be questionable in my mind just exactly how one would proceed. I am wondering about the film base. You see, you could use quartz perhaps as a film base, but I doubt if you would be free from some chemical reaction between the quartz and the platinum or the rhodium if you observe them at such high temperatures.

A. T. FELLOWS:[5] Was it completely established or was there something of an assumption that the film that you are measuring the reflectivity of had the same properties as these check films on either side? I am thinking of that all-pervading "grease" that might be on the glass slides in one case and possibly not in another. After all, you are measuring the thickness of a film on one slide and saying that is the thickness of a film on another slide, are you not?

MR. COLEMAN: That is right. You see, the basis of that measurement is that the surface density of deposits is constant, and to test that we put a bunch

[4]President, Harold E. Trent Co., Philadelphia.

[5]Research chemist, Metals Disintegrating Co., Elizabeth, N. J.

of small section slides at various places and determined the limits of variation of surface density. The method of cleaning the slides is exactly the same as you would prepare a mirror for coating it with aluminum, and we believe it would be free from any "grease".

MR. FELLOWS: You would say the films are the same?

MR. COLEMAN: Oh, yes. There is only one question, and that is whether or not the crystal structure may be different in a film which is condensed as compared with bulk metal. I think that there have been some papers and work listed in Barrow's Diffusion Book, "Diffusion of Solids", from which it appears that at the temperatures in question there is no need to be concerned with the crystal structure problem.

W. R. HAM: I might bring out a point that Dr. Coleman hasn't brought out with regard to the check on the thickness. A further check on the determination of the thickness of the films used would be to add the weight of the control slides which should be approximately equal to the weight of the bi-metallic film on the slide used to observe the diffusion. If the sum of the thicknesses of the two control slides should be equal to the total thickness of the bi-metallic film with reasonable precision, then it would be evident that the surface density of the deposits will be nearly constant.

MR. COLEMAN: Yes, we did do that.

MR. HAM: I presume they had, but I hadn't seen that.

MR. COLEMAN: We did add up the weights of both control slides and found that that was equal to the weight of the bi-metallic film on the center slide.

J. E. MILLEN:[6] I notice the authors used the only two colored metals in their investigation: copper and gold. Would that method suffice where you have two metals that are of similar colors, such as lead and tin?

MR. COLEMAN: The question has come up as to why we used gold and copper as contrasted with zinc and lead. Of course, they are different colors and we comment on that specifically in the paper, but the thing is that The Method of Thin Films can be used for any bi-metallic systems or more complicated systems, for that matter, by just shortening the wave length. One does not have to use visible light. As a matter of fact, we plan to use X-ray reflection and electron diffraction methods in exactly the same fashion as visible light used here. In other words, we are shortening the wave length for such cases. I think as a result of preliminary experiments, even the reflectivity change is enough, but to make the method really marked, one would want to use shorter wave lengths than visible.

Authors' Reply

Just very briefly we might say that in the paper proper, at the end in order to answer such questions as these that we have taken up, we have thrown in some things called "Comments on the Method of Thin Films". In this section, all these points except Dr. Ham's point of using emissivity, we considered but the latter was omitted by oversight. Dean Whitmore indicates something that is not emphasized in the paper; that is, that constitution diagrams may be

[6]Research laboratory, American Works, American Steel & Wire Co., Cleveland.

constructed using the reflectivity of homogenized samples. This would be done by plotting the reflectivity of the homogenized sample as the function of its composition. There should be at least a correlation between the ordinary constitution diagrams and the composition-reflectivity curves.

Another thing Dean Whitmore mentions is that gaseous diffusion is observed by The Method of Thin Films. If you heat a copper slide in a vacuum reflectometer in the presence of oxygen at a known pressure, one could tell exactly when the first oxygen atoms had completely penetrated the film by making the reflection through the glass slide; the glass slide, of course, would prevent the oxygen from getting into that place, and then you would be measuring the invasion, so to speak, i.e., the time required for the oxygen to actually pass completely through the copper film. He further points out that certain thermodynamic quantities such as dissociation pressures could be determined by noting the rate at which the oxide decomposed. The temperature and pressure at which the film disappeared would give values of certain oxides. Velocity constants, heats of formation, and specific heats, could be determined by using the reflectivity curves as reaction rate curves. That is quite an undertaking, but presumably there is no reason why it could not be done. Dr. Turner mentions that The Method of Thin Films could be used in studying the deterioration of metallized mirrors which, of course, is very important right now. Some work has been done by us in this connection, but no conclusive results have been obtained as yet.

NOTCHED BAR TENSILE TESTS ON HEAT TREATED LOW ALLOY STEELS

By G. Sachs and J. D. Lubahn

Abstract

Various oil-hardening alloy steels of the 0.40 per cent carbon class were tested as notched tensile test bars in various conditions of strength level as determined by the tempering temperature.

It was found that the ductility of the steel was considerably reduced in the notched section, but that the notch strength was not impaired as long as the steel had an ultimate strength below 200,000 pounds per square inch. For such soft steels, the strength was increased by a percentage which was approximately equal to the percentage cross-sectional area removed by the notch. For stronger steels, a shallow notch did not seriously affect the notch strength and notch ductility. However, the ductility of deeply notched specimens was nearly zero and their strength greatly reduced. This condition prevailed for steels with an ultimate strength of between 220,000 and 280,000 pounds per square inch, although the ductility given by ordinary tensile tests remained almost as high in this strength region as for the softer steels. Thus the values found from an ordinary tensile test are not a sufficient criterion of the suitability of a steel for commercial applications involving shoulders, keyways, and other irregularities in shape that produce a similar effect to that of a deep notch.

The embrittling effect of factors such as overheating and large section size is not shown by ordinary tensile tests, but it is readily observed from the results of notched bar tensile tests.

An attempt has been made to correlate the results with the shape of the stress-strain curve for the notched section, and with previous conceptions of the laws of plastic flow and the stress distribution in a notched section.

This paper is a report on part of the research program conducted at Case School of Applied Science under the sponsorship of the International Nickel Company. Appreciation is expressed for the co-operation of Mr. P. Stefan, now with Chase Brass and Copper Company, who conducted a considerable number of the tests on notched tensile test bars.

A paper presented before the Twenty-fourth Annual Convention of the Society held in Cleveland, October 12 to 16, 1942. Of the authors, G. Sachs is professor of physical metallurgy, and J. D. Lubahn is research assistant, department of metallurgical engineering, Case School of Applied Science, Cleveland. Manuscript received May 9, 1942.

MANY different cases have been reported in which steels and other structural metals break in a brittle manner. Such a fracture can usually be distinguished either by its appearance, which is characteristically different for brittle and for ductile conditions, or by the lack of a measurable strain at the point of failure.

However, excluding the thoroughly investigated conditions which are conducive to failure under vibrational and impact loads, little experimental evidence is available regarding other types of brittle ruptures. While considerable literature has been published on this subject, the conclusions drawn from this work are very controversial. In particular, a test is desired in which the stress state can be varied sufficiently to cause either ductile or brittle failures of a steel which is normally ductile, but which might behave in a brittle manner under extreme service conditions characterized by a stress state particularly conducive to brittleness.

It is generally believed that the addition of "transverse" stresses in both directions perpendicular to a major applied (or "longitudinal") tension, resulting in a "triaxial tension", will reduce the ductility of the metal. Any metal should become brittle if the triaxiality becomes sufficiently great, i.e., if there is a sufficiently close approach to a state of "hydrostatic tension", in which the transverse tensile stresses equal the longitudinal stress.

A considerable amount of research has been carried out on the effect of notches on the behavior of steels and other metals subjected to static tension. However, it appears that the interpretation of the experimental results which are available at present is rather inconclusive and open to argument.[1]

Therefore, notched bar tensile tests on several structural steels, heat treated to various strength levels, were conducted. These yielded a number of fundamental facts which apparently have not been previously recognized. Also, an attempt will be made to correlate the experimental results with general metallurgical conceptions regarding the plastic flow of metals (1).[2]

Material and Heat Treating

For the experimental investigation of the properties of notched bars, five open-hearth, ladle-killed alloy steels of the 0.40 per cent

[1] A rather complete bibliography on this subject is appended. The results of the previous investigations are discussed critically elsewhere (51).

[2] The figures appearing in parentheses refer to the bibliography appended to this paper.

carbon class were selected. The chemical compositions of these steels are given in Table I.

Table I

	S.A.E. No.	Chemical Composition, Per Cent C	Ni	Cr	Mo	Mn	P	S	Si
A	2340	0.39	3.37	....	....	0.78	0.028	0.017	0.24
B	3140	0.40	1.37	0.66	....	0.76	0.020	0.015	0.26
C	4140	0.38	....	1.00	0.19	0.80	0.024	0.025	0.23
D	5140	0.40	....	1.00	....	0.69	0.014	0.023	0.16
E	T-1340	0.42	....	....	....	1.81	0.023	0.022	0.23

This report deals mainly with the results obtained on the S.A.E. 2340 steel. The other steels yielded fundamentally the same relations, although some complex phenomena in both the regular and the notched bar tensile tests were observed in these steels which have not been completely clarified as yet.

The following products were processed for the investigation: hot-rolled rod, ¾, 1½ and 2¾ inches in diameter, and seamless tubing, pierced and cold-drawn from 2¾-inch diameter billets to $1\frac{23}{32}$ inches outside diameter and ⅛ inch wall thickness, normalized, and drawn at the A_1 point.

Suitable lengths of rod were cut off and prepared for the notched bar tensile tests by the following procedure: (a) standardizing heat treating, (b) preliminary machining, (c) quenching and tempering, (d) final machining, and (e) measurement of dimensions.

The standardizing heat treatment consisted of: (a) heating for one-half hour at 1650 to 1700 degrees Fahr. (900 to 925 degrees Cent.) in a Hevi Duty Furnace, (b) air cooling down to 700 degrees Fahr. (370 degrees Cent.) or lower, the specimens being placed individually on a transite board, (c) tempering for 4 hours at a temperature of 1175 to 1200 degrees Fahr. (635 to 650 degrees Cent.) in an electric Lindberg Cyclone forced convection furnace, and (d) furnace cool.

In order to secure a rapid and uniform quenching, special heat treating equipment was built, Fig. 1. It consisted of a vertical, controlled atmosphere furnace with a sliding trap door from which the specimen could be dropped into an oil tank. The approximate critical points and quenching temperatures[3] for the various steels are given in Table II.

[3]These somewhat arbitrary quenching temperatures were selected in order to permit the successive heat treating of the numerous specimens without frequent changes of temperature.

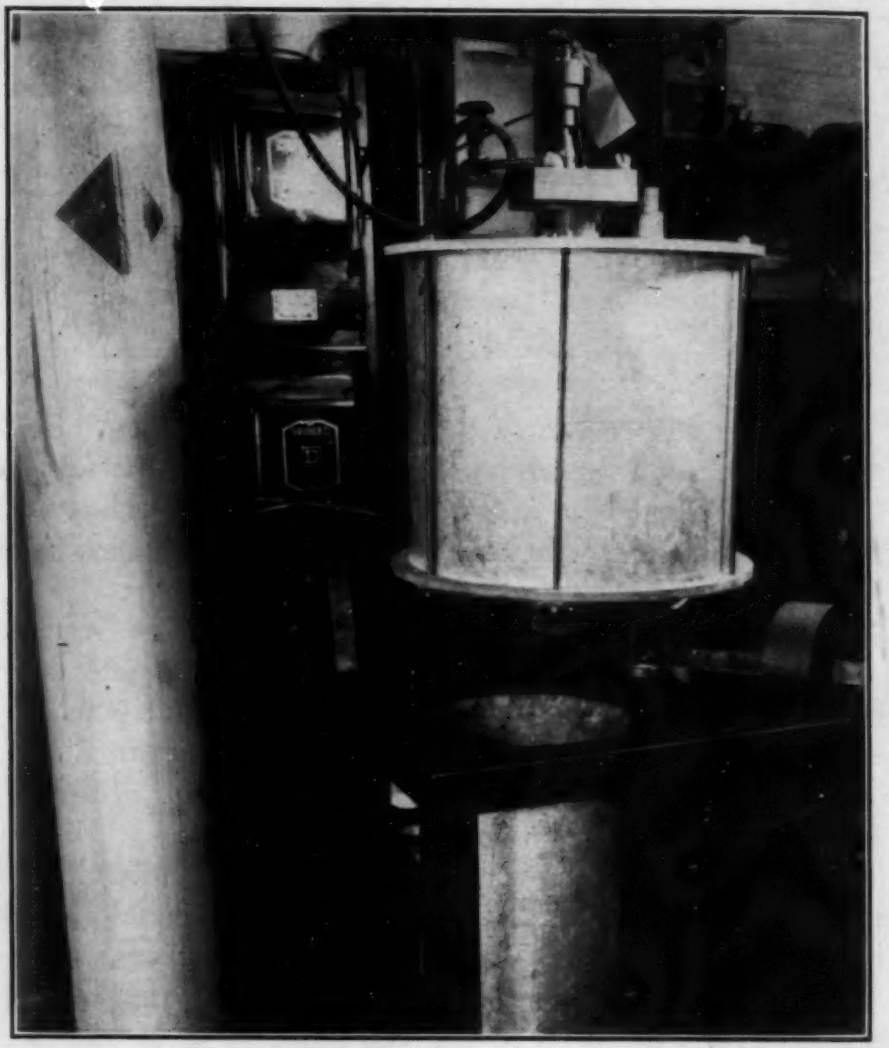


Fig. 1—Controlled Atmosphere Furnace and Air Lift Quenching Tank for Hardening the Specimens.

In the first series of experiments, covering approximately one-half of the results, hydrogen was used as a controlled atmosphere. However, while the expected hardness values were obtained in the beginning, indicating a pure condition of hydrogen, later experiments yielded low surface hardness.[4] Therefore, hydrogen was abandoned

Table II

Steel:	S.A.E. 2340	S.A.E. 3140	S.A.E. 4140	S.A.E. 5140	S.A.E. T-1340
Critical Point (Ac_3)	1360° F.	1450° F.	1485° F.	1440° F.	1435° F.
Quenching Temperature	1450° F.	1600° F.	1600° F.	1600° F.	1600° F.

[4]Very heavy decarburization, associated with cracking on quenching, was generally found to occur on hardening at temperatures of 1800 degrees Fahr. and higher in hydrogen.

and replaced by forming gas (92 per cent N, 8 per cent H). Slight decarburization was obtained with this gas mixture at the regular hardening temperatures.

The quenching tank, Fig. 1, consisted of an open cylindrical shell within a cylindrical container, forming an inside and outside chamber. Three air lifts in the outer chamber gave the oil an upward motion in this part and a downward motion around the specimen in the inner chamber. The tank contained a total volume of soluble quenching oil[5] of about 10 gallons, and with this volume of oil, the temperature could be kept between 90 and 120 degrees Fahr. (31 and 50 degrees Cent.). A basket, with large holes in the bottom, was provided for keeping the specimen off the bottom of the tank during quenching and for removing the specimen after quenching.

The specimens were tempered for 30 minutes in an electric air-convection type furnace which gives fast heating and uniform temperature distribution.

Regular Tensile Tests

From the specimens softened by the standardizing heat treatment, tensile test specimens were machined, Figs. 2a and b.

Special holders with ball seats, Fig. 3, for the threaded end specimens were made from heat treated S.A.E. 3140 steel, following in general the recommendations of the A.S.T.M. specifications E8-35. However, it was impossible to avoid Brinelling of the flat surfaces on which the 1-inch diameter balls rested, and free movement was restricted even if this device was used.[6] Therefore, it was preferred to place the steel balls on soft and lubricated copper pads, assuming that in this case there would be less friction than when the ball is in direct contact with the impression it would make for itself in the steel holder.

During the tests, the diameter changes of a number of specimens were followed up by means of a micrometer, and the "yield strength" determined graphically as the stress corresponding to a displacement from the elastic straight line of 0.1 per cent of the diameter, thus corresponding to 0.2 per cent permanent elongation. Maximum load

[5]We are indebted to Mr. C. L. Johnson, Franklin Oil and Gas Company, for the recommendation and prompt delivery of this oil.

[6]A few experiments, using strain gages on two sides of the specimen, have shown that the tensile stress on one side might be 50 per cent higher than that on the other side. In order to avoid such an additional bending which is particularly objectionable in testing the notched bars, a new testing fixture is being built at the present time.

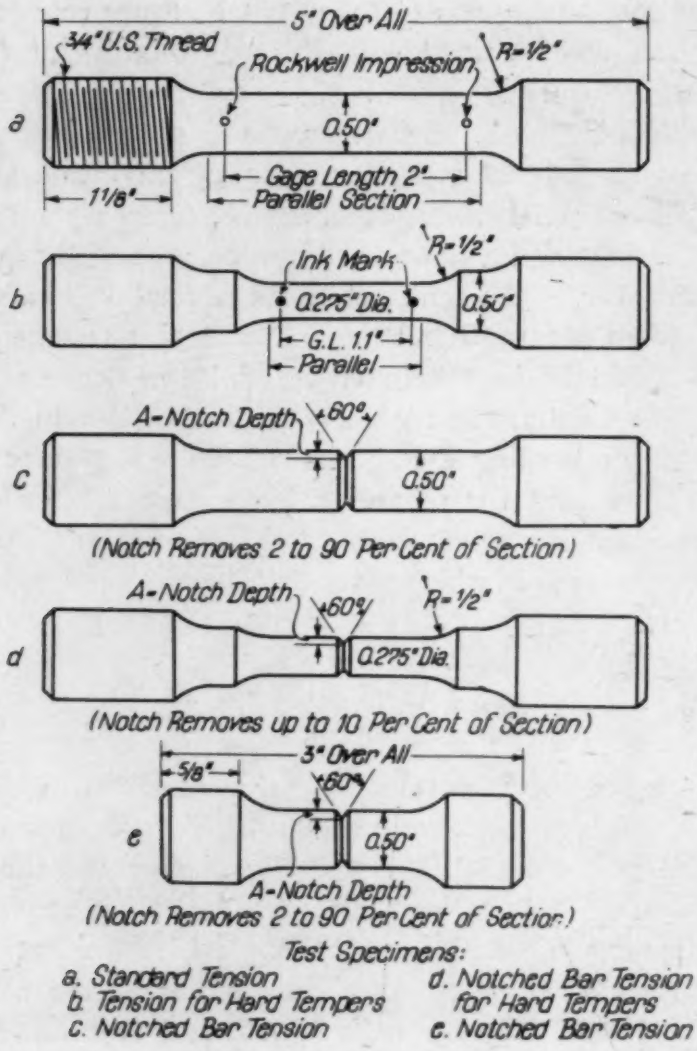


Fig. 2—Test Specimens.

and breaking load were recorded. The maximum load divided by the original cross-sectional area represents the "ultimate strength", while the breaking load divided by the cross-sectional area after failure was taken as the "fracture stress." The "elongation" of unnotched bars was measured over a gage length of 4 times the diameter. The "contraction in area" or reduction of cross-sectional area after failure was determined from measurements of the diameter after failure by means of a measuring microscope. The "uniform reduction" was calculated as the change in cross-sectional area some distance apart from the failure where the section was fairly constant over a certain length.

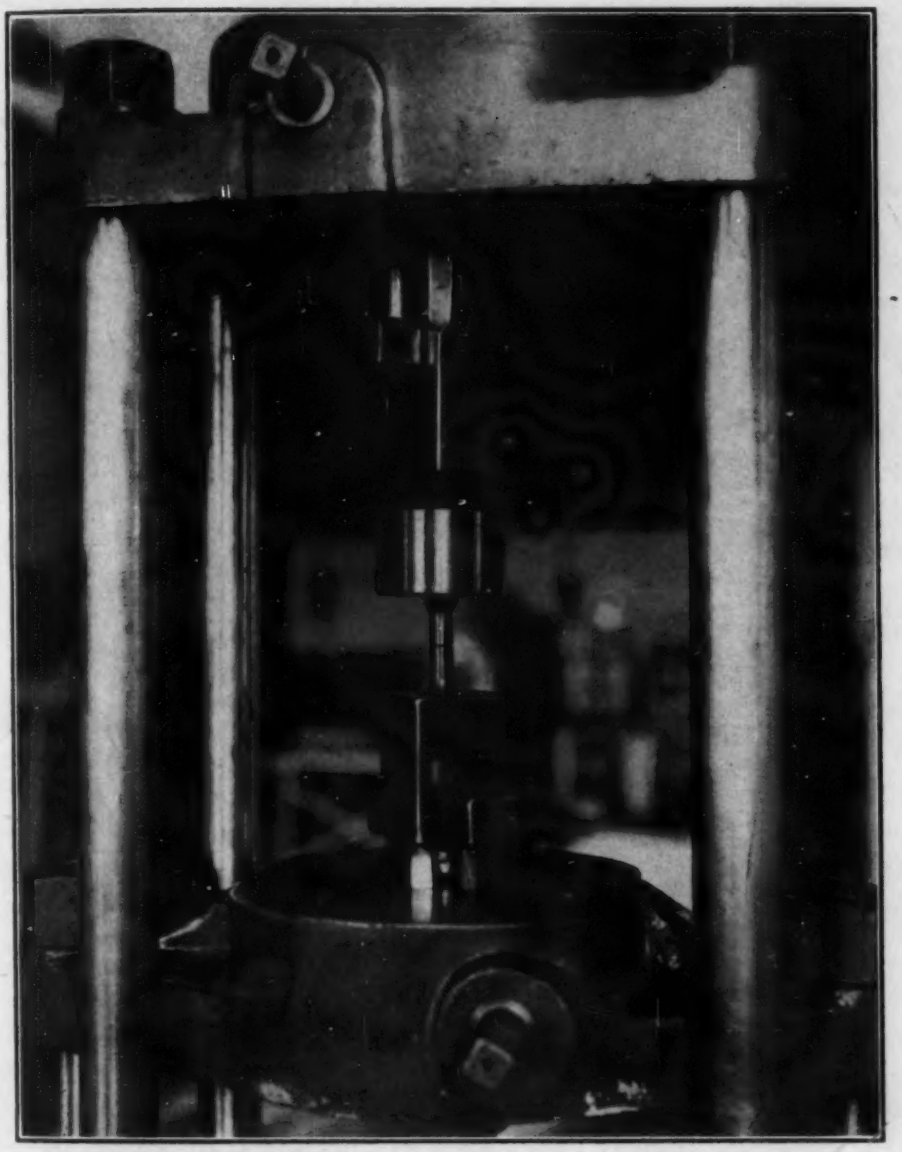


Fig. 3—Self-Aligning Tensile Test Fixtures and Specimen Under Test.

Results of the tests on unnotched specimens for the S.A.E. 2340 steel are presented in Fig. 4.

The very regular curves for the various tensile test characteristics can be considered as a proof that both the chemical composition of the steel and the procedure of heat treating were as uniform as could be expected. The ultimate strength, the variations of which are a good measure of uniformity, did not deviate more than $\pm$ 3 per cent from a smooth trend curve if plotted versus the tempering temperature.

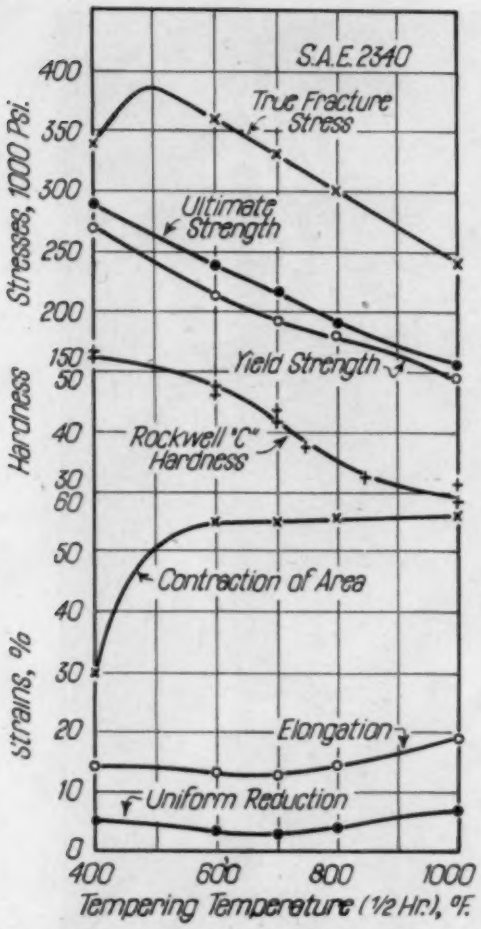


Fig. 4—Results of Regular Tensile Tests on a Low Alloy Steel.

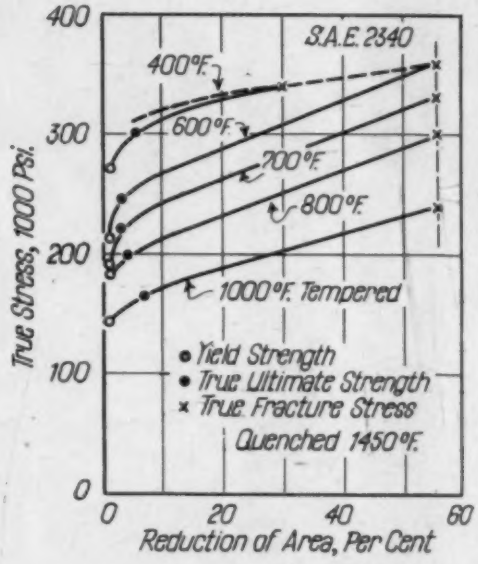


Fig. 5—True Stress-Strain Curves for S.A.E. 2340 Steel Tensile Tests, Heat Treated to Various Strength Levels.

The various characteristics of the tensile tests correspond quantitatively to the previously observed properties of such alloys (45), (46). However, it was observed that specimens of all alloys hardened and tempered to an ultimate strength up to approximately 260,000 pounds per square inch possessed a contraction in area of 50 per cent or higher.

These tensile test characteristics point to a rather general misconception regarding the suitability of a steel for practical applications. It is generally believed that a steel becomes increasingly valuable if the strength can be increased without serious loss in ductility, and a regular tensile test is the most common means of evaluating these properties. If this were true, then the S.A.E. 2340 steel, the true stress-strain curves of which are represented approximately in Fig. 5, would be most suitable for high strength applications when

tempered at a temperature of approximately 600 degrees Fahr. (315 degrees Cent.), resulting in the most favorable combination of high strength and high ductility.

However, practical experience indicates that any steel becomes rather sensitive and prone to premature failure in service, particularly in structural applications which involve section changes and impact loads, if heat treated above a certain strength level. The general decrease of the impact values with decreasing tempering temperature (45) or increasing strength level (see Fig. 20) is considered as a manifestation and confirmation of such a relation, and the minimum of the impact strength at 600 degrees Fahr. (315 degrees Cent.) indicates particularly poor characteristics when tempered at this tempering temperature. Thus, the performance of a steel in practice, which as a rule involves stress raisers and sudden loads, cannot be judged by the characteristics as measured by regular tensile tests, the values of contraction in area apparently being particularly misleading.

Notched Bar Tensile Tests

Test Procedure—Notched specimens, Fig. 2c, d, and e, were provided with a circular, very sharp V-shaped groove in the center of the cylindrical portion. The notches had an included angle of 60 degrees and a varying depth, removing anywhere from approximately 2 to 90 per cent of the cross-sectional area. The diameter of the cylindrical part was, as a rule, 0.500 inch, but it was made 0.275 inch for specimens having a notch smaller than 10 per cent and tempered at a temperature of 600 degrees Fahr. (315 degrees Cent.) or less in order to avoid thread breaks. The total length of the specimens was either 5 or 3 inches.

Most of the specimens were notched before heat treating. The notches were cut with a cobalt high speed steel tool leaving an unavoidable small fillet of varying radius at the bottom of the notch. In order to obtain a really "sharp" notch, most of the specimens were finished after heat treating with an equilateral file which had been ground smooth on two sides, forming a sharp 60-degree wedge, as suggested by Gensamer (24). This procedure gave a sharp, V-shaped notch, Fig. 6, except for the very hard tempers, above 50 Rockwell "C", in which case the notch remained irregular and usually showed a sharp dent in the bottom of the fillet.

The most information was expected from a stress-strain curve for the notched section. Such a stress-strain relation can be obtained by plotting the force divided by the initial cross-sectional area of the notched section, or conventional stress, versus the reduction in cross-sectional area of the notched section. Both the stress and the strain are only averages of values which might vary considerably over the cross section, and their physical meaning has

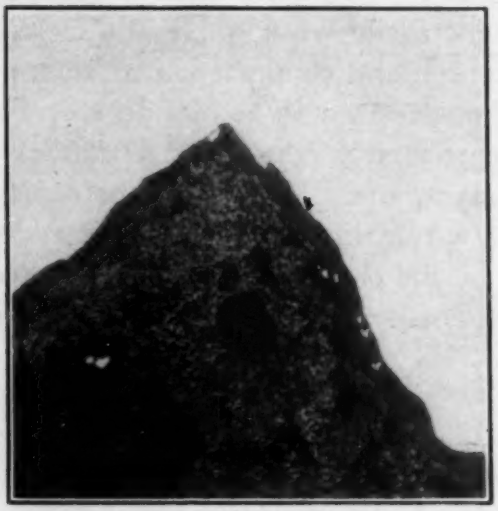


Fig. 6—Longitudinal Section (× 200) Through a Tensile Specimen, Showing the Sharpness of the Notch as Produced by Finishing With a File Which Has Been Ground Smooth on Two Sides.

not been disclosed as yet. For specimens having a measurable ductility, such stress-strain curves were obtained in the following manner: For a given load (P), the diameter (D) of the unnotched section was determined with a regular micrometer, and an average diameter of notched and unnotched section (d) by means of a micrometer having one chisel-shaped anvil with an angle of less than 60 degrees. From such readings the "reduction of area" (R) is calculated by the following equation:

$$R = \frac{4\,(d_o - d_1) - 2\,(D_o - D_1)}{2d_o - D_o},$$

where d_o and d_1 are the readings with the chisel anvil micrometer (or distances from the bottom of the notch to the opposite surface) and D_o and D_1 are the diameters of the unnotched section for the unstrained and strained condition respectively. (It was observed that the diameter of the unnotched section adjacent to the notch also

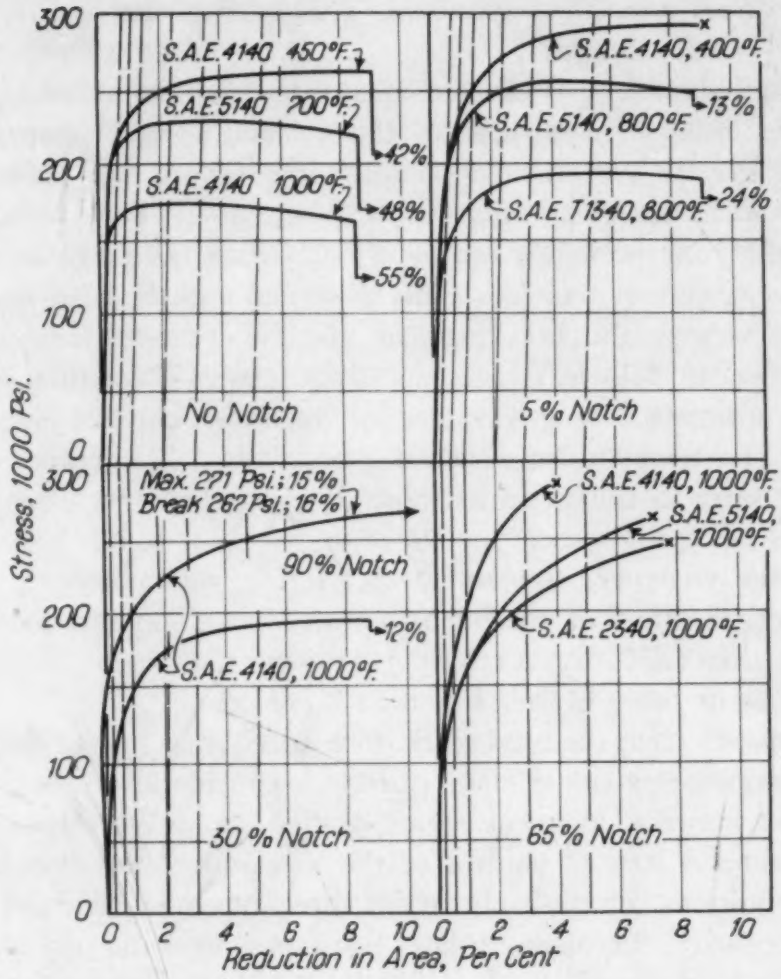


Fig. 7—Stress-Strain Curves for a Number of Selected Unnotched and Notched Bars.

decreased during the tests, particularly for specimens having a shallow notch.) The stress (S) is given by the equation:

$$S = \frac{4P}{\pi\,(2d_o - D_o)^2}.$$

A number of stress-strain curves obtained in this manner are represented in Fig. 7.[7]

The maximum load divided by the original cross-sectional area of the notched section yields a value corresponding to the ultimate

[7]These curves are not very accurate, particularly for soft specimens, as the chisel shaped anvil caused a slight indentation. By taking repeated measurements at zero load, a correction for this wear could be obtained. Thus, stress-strain curves were developed which qualitatively illustrate the effects of notching, but which must be repeated using an accurate strain gage in order to yield reliable, quantitative relations. The construction of such a gage is in progress.

strength of an unnotched specimen. This average strength is designated as "notch strength".

"Notch ductility" is defined as the contraction in area, or reduction of cross-sectional area of the notched section after failure. The diameter of the notched section after failure was determined by means of a measuring microscope.

In tests on specimens having a soft temper, failure at a load below the maximum was frequently observed and the load recorded. The ratio between this breaking load and the cross-sectional area of the notch after failure is introduced as (true) "fracture stress".

The ultimate strength values of identical notched specimens tempered at a temperature of 800 degrees Fahr. (425 degrees Cent.) or higher were as consistent as those of unnotched specimens, other conditions being the same.

On the contrary, tremendous scattering was observed in the notch strength values of specimens tempered at a temperature of 700 degrees Fahr. (370 degrees Cent.) or less, the notch ductility of such specimens being always less than 2 per cent.

It appears from the total work done so far that the scattering of values obtained by a single series of tests is considerably less than the differences between identical series carried out at different times. This indicates a lack of control of the machining, heat treating, or testing conditions, or probably of all three types of conditions. It will be necessary, therefore, in later tests to control the procedure to a much greater degree than is possible with the equipment used at the present time. This also requires the development of a measuring technique for small strains in the transverse direction. The experimentation is being continued along such fundamental lines as mentioned above.

In spite of these difficulties, the tests performed so far reveal a number of hitherto unknown relations regarding the notch strength of heat treated alloy steels. While the quantitative values will be open to considerable argument, the qualitative results obtained reveal a picture of the notch effects which is in agreement with theoretical conceptions.

General Relation Between Notch Strength and Notch Ductility—The notch strength is dependent upon numerous factors such as, particularly:

(a) the chemical composition of the steel,

(b) the ultimate strength or "strength level" of the steel, and

(c) The shape of the notch as determined by depth, notch angle, and bottom radius.

However, for a notch of given shape, the ratio between notch strength and ultimate strength has been found frequently to be approximately the same, and to be rather close to 2 to 1 for a notch of extreme depth (100 per cent) and sharpness. Furthermore, a general survey of the data presented has revealed the fact that this

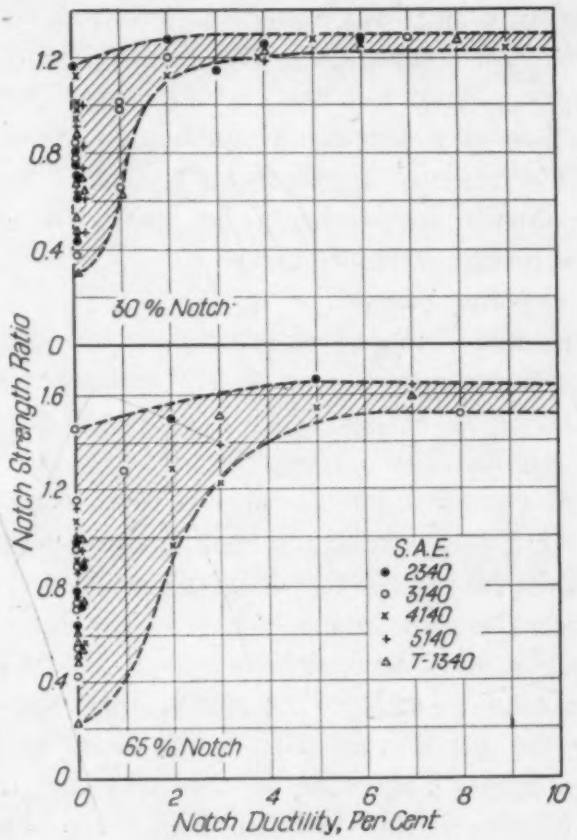


Fig. 8—Relation Between Notch Strength Ratio and Notch Ductility.

notch strength ratio, for a given notch, depends only upon the notch ductility as illustrated in Fig. 8 for the specimens having a 30 per cent and a 65 per cent notch.

While the physical meaning of the "notch ductility" has not been adequately explained as yet, both previous and new experimental data indicate that the notch ductility is that value obtainable in the notched bar tensile tests which is subject to the most fundamental generalizations. The complex behavior of other properties

is partially explained by the simpler variations of the notch ductility.

Fig. 8 shows clearly two different ranges regarding the notch strength ratio.

Bars having a notch ductility above a certain value, which is approximately 5 per cent for all the investigated steels, possess a practically constant notch strength ratio. This ratio is 1.25 $\pm$ 0.05 for such "ductile" specimens with a 30 per cent notch and 1.6 $\pm$ 0.05 for specimens with a 65 per cent notch. The scattering of the values is small, indicating a uniform condition which is little affected by variations in chemical composition, heat treating, and testing.

On the contrary, "notch brittle" bars having a notch ductility of 1 per cent or less may possess a notch strength ratio anywhere between a very low value of approximately 0.2 to 1 and a value close to that for the ductile specimens. The source of this tremendous scattering of the notch strength ratios for such notch brittle steels cannot be fully explained as yet.

In the intermediate range between the ductile and brittle ranges, the notch strength ratio decreases slowly with decreasing ductility.

If the scattering is disregarded, the shape of the notch strength ratio versus notch ductility curves, Fig. 8, has the appearance of conventional stress-strain curves. This is more than a mere superficial similarity, but has a real and fundamental significance. The stress-strain curves in Fig. 7, for different steels and different temperatures, but for the same notch, are quite similar. If the curves for various ductile specimens are plotted in such a manner that their maximum load is selected as unity, they appear sufficiently alike to justify the simplifying assumption that for a given notch the stress-strain curves converted in this way are identical. This approximate similarity is illustrated in Fig. 9 for specimens having a 5 per cent notch,[8] this being the only depth of notch yielding a large number of ductile specimens. If the same procedure is applied to a few stress-strain curves for specimens having a 65 per cent notch, Fig. 7, the results can be combined to give a qualitative picture of the approximate shape of such an average derived stress-strain curve for this notch, Fig. 10 (insert). Now, considering this curve as basically identical for any combination of composition or tempering temperature which has been investigated, a set of curves can be constructed illustrating the as yet unknown shape of

[8]Actually, in these stress-strain curves of notched and unnotched specimens, the knee becomes sharper with increasing tempering temperature.

actual stress-strain curves for specimens having various, and particularly low, ductilities. This is illustrated in Fig. 10 for the S.A.E. 2340 steel specimens having a 65 per cent notch. The universal stress-strain curve, Fig. 10 (insert), is converted into individual curves, Fig. 10, by multiplying its ordinates by 1.65 times the ultimate strength of the unnotched specimen which was tempered

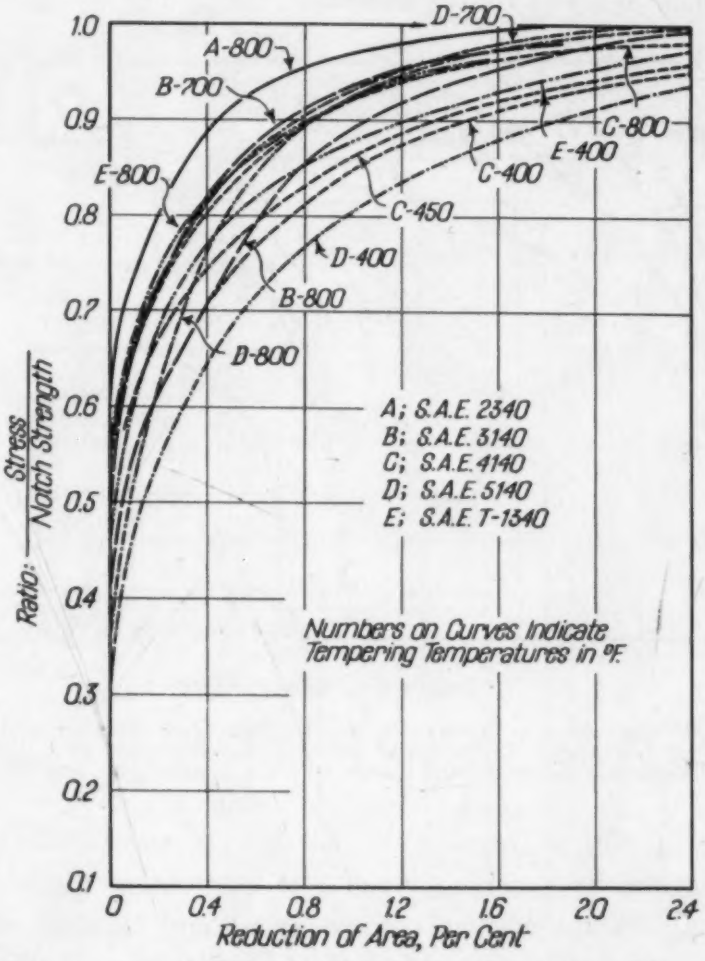


Fig. 9—Derived Stress-Strain Curves for 5 Per Cent Notched Specimens Made from Various Steels and Tempered at Various Temperatures.

at the same temperature, see Fig. 4. In other words, the resulting curves would represent regular stress-strain curves, but for specimens possessing sufficient ductility to reach a maximum, which is assumed to be 1.65 times the ultimate strength. Then each curve is terminated by a point having an ordinate corresponding to the observed notch strength. It is now readily explained that by reversing the process, these terminating points would define the universal

stress-strain curve for the particular notch considered. This curve should be identical with the composite notch strength ratio versus notch ductility curve in Fig. 8. Such a conclusion cannot be definitely proved or disproved because of the large scattering of the experimental results, but the agreement appears sufficient to justify the use of this conception, which will later be made the basis of a more complete analysis of the results.

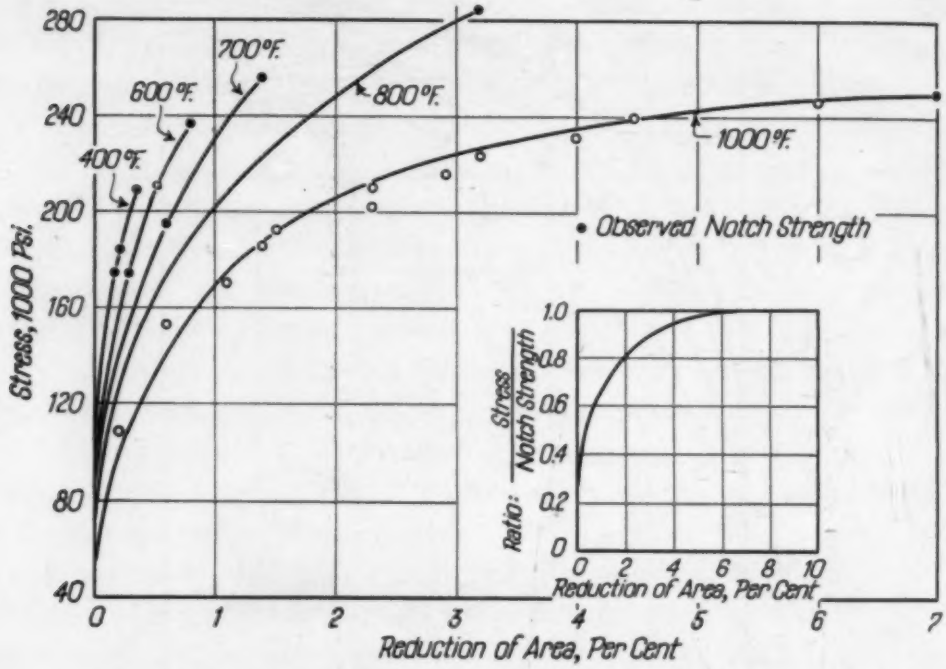


Fig. 10—Stress-Strain Curves for S.A.E. 2340 Steel Specimens With a 65 Per Cent Notch and Tempered at Various Temperatures as Constructed from the Universal Stress-Strain Curve for this Notch (See Insert).

Effects of Notch Depth—The changes in the tensile test characteristics caused by notching cannot be adequately explained without a thorough knowledge of the external and internal effects introduced by variations in notch depth. Therefore, this phase of the program has been investigated in a particularly extensive manner. Nevertheless, the results of tests on the different steels heat treated to various strength levels, as represented in Fig. 11 for the S.A.E. 2340 steel, are still not quite conclusive.

Again, the notch ductility appears to be the property which is subject to the simplest relations.

The notch ductility versus notch depth curves for conditions where the ductility is measurable over a considerable range of notch depth, Fig. 12, illustrate the universal effect of notch depth on

notch ductility. The notch ductility decreases with increasing notch depth, at first rapidly, then more and more slowly, and apparently reaches a minimum value at a notch depth of approximately 50 per cent. The upward trend of the curves in the region of very deep

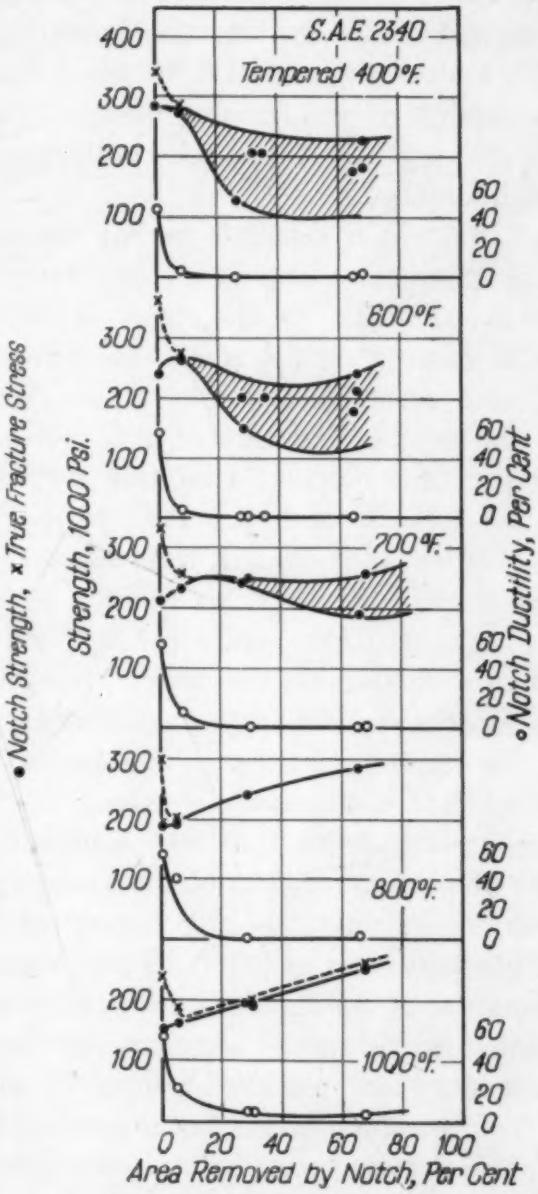


Fig. 11—Effect of Notch Depth on Notch Tensile Test Characteristics of Low Alloy Steel.

notches is not fully substantiated by the results represented here, but agrees well with previous results (4).

For a moderately deep notch many of the conditions of com-

position and tempering temperature which have been investigated cause complete brittleness, i.e., a contraction in area smaller than 1 per cent, see Fig. 12. In all these instances, a further increase of notch depth does not cause any noticeable increase of ductility again.

The ductility, or contraction in area, of unnotched specimens apparently has little relation to the notch ductility of deeply notched bars. For any of the steels investigated, the notch ductility decreases with increasing strength level and becomes practically zero at strength levels for which there is still a very high contraction in area if the specimen is tested unnotched.

The shape of the notch strength versus notch depth curves, Fig. 11, undergoes a radical change with increasing strength level, or decreasing notch ductility. If the notch ductility of a deeply notched specimen is above a certain small value, approximately 2 to 3 per cent, the notch strength increases almost linearly with the area removed by the notch. The rate of this increase is such that the limiting, 100 per cent notched, condition would have a notch strength between 1.7 and 2.0 times the ultimate strength of the unnotched specimen. Alloy and strength level have a slight effect on this limiting notch strength ratio. This relation is limited to steels having a strength below 210,000 pounds per square inch.

For notch brittle conditions, the notch strength versus notch depth curves have not been definitely established as yet, because of the scattering of the results. It appears that the notching of steels having a high strength level changes the strength little at first, but then, with increasing notch depth, the large scattering is introduced. The highest values of notch strength obtained might be considerably higher than the ultimate strength of the unnotched specimen while the lower limit might be as low as 20 to 30 per cent of the ultimate strength. The boundaries of the scattering region of the notch strength versus notch depth curves are wavy in nature, the upper limit being confined to a rather narrow strength range and the lower limit descending from the high value for unnotched specimens to a low level for deeply notched specimens.

An attempt can be made to establish a theoretical trend of notch strength versus notch depth curves from the experimentally determined stress-strain curves, Fig. 7, by taking advantage of the assumption which was previously proposed. For each depth of notch, a universal stress-strain curve can be constructed, as illustrated in Figs. 9 and 10. However, for the sake of comparing dif-

ferent notch depths, the ultimate strength of the corresponding unnotched specimen will be considered as the unit of the ordinate scale rather than the maximum load point (real or fictitious) of the stress-strain curve for a notched specimen. Then in this representation the stress, for a given strain, will be some fraction of the ultimate strength of the unnotched specimen. Now a family of curves can be plotted, where each curve shows the derived stress as a function

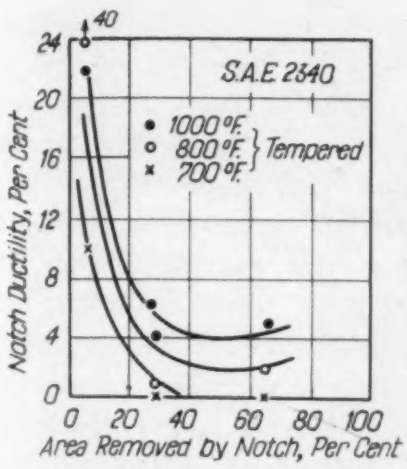


Fig. 12—Effect of Notch Depth on Notch Ductility of a Low Alloy Steel.

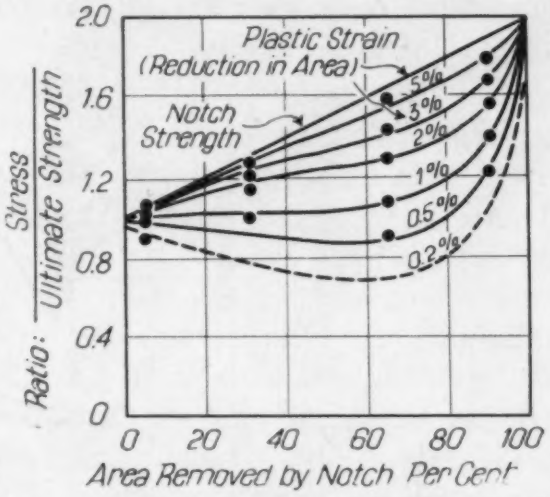


Fig. 13—Composite Chart of the Stresses Required to Strain by Various Amounts Notch Tensile Bars Having Notches of Various Depths (Ordinate Gives Ratio Between Stress and Ultimate Strength of Unnotched Metals).

of notch depth for some particular strain and where different curves correspond to different strains. Such a family of curves is shown in Fig. 13 for strains of 5, 3, 2, 1, 0.5, and 0.2 per cent. A series of such curves is not particularly accurate, but it gives an approximate composite diagram of the behavior of ductile and semi-brittle notched specimens. Actually, these curves must be derived from very ductile specimens, tempered at high temperatures (800 to 1100 degrees Fahr.), and they might be somewhat different for lower tempering temperatures.

Such a diagram, Fig. 13, now permits the construction of a notch strength ratio versus notch depth curve for a given curve of notch ductility versus notch depth, Fig. 14. With notch ductilities which are above approximately 5 per cent for all notch depths, curve (a) in Fig. 14, the notch strength assumes the highest possible values. It may be assumed, for reasons of simplicity, that this maximum notch strength versus notch depth curve approaches a straight line

with a limiting notch strength ratio of 2, as previously discussed. A scattering of ± 1 per cent in the notch ductility, curve (a) in Fig. 14, will not materially affect the trend of the notch strength curve (a′). This condition, however, is radically changed if the notch ductility decreases and becomes, for instance, as low as 1.5 per cent for deeply notched specimens, scattering between say 0.5 and 2.5 per cent, curve (b). The corresponding notch strength values are now represented by a wide shaded area (b′), spreading fanlike from a

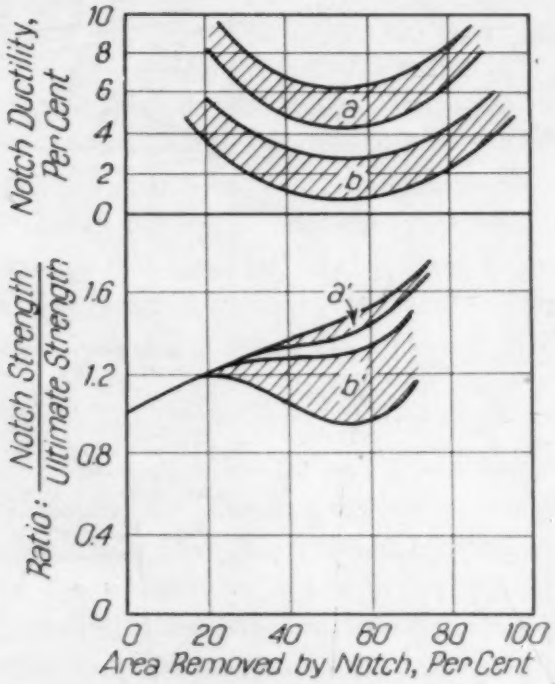


Fig. 14—Diagrammatic Representation of Correlation Between Notch Ductility (a, b) and Notch Strength (a′, b′) for Various Notch Depths.

point somewhere on the maximum curve close to the point of zero notch. The lower the temperature and hence the lower the notch ductility also, the larger is the shaded area representing the scattering area, and the smaller is the notch which is required to produce scattering of the results. These relations, while not established accurately, readily explain the increasing scattering of notch strength, both with decreasing notch ductility and with increasing notch depth.

The peculiar shape of the curves representing the stress for various strains versus the notch depth in Fig. 13 results from the shape of the stress-strain curves for specimens having a notch of interme-

diate depth. These curves deviate from the elastic line more gradually and have a less sharp "knee" than the stress-strain curves both for an unnotched bar and for a very deeply notched bar. This behavior has been previously observed (4), (17), and has been ascribed to the stress concentration which is present in a notched bar during elastic straining and which is gradually eliminated by plastic strain. However, it was concluded from the earlier experiments that this effect of stress concentration is eliminated by a plastic strain of approximately 0.5 per cent (17), (18), since the stress for a strain of 0.5 per cent was found to be proportional to the notch strength for all the notch depths which were investigated. This, however, is certainly not true for the steels which have been investigated here, for it can be seen that the ordinates of the curves in Fig. 13 are not proportional to the notch strength, but that a curvature is retained even for strains close to the maximum load strain. This curvature decreases considerably with increasing strain, indicating a partial but not complete relief of stress concentration by plastic straining. The minimum in the curve representing stress versus notch depth for the smallest strain which could be determined with reasonable accuracy (0.2 per cent) leads to the conclusion that the stress concentration is a maximum for a notch depth of approximately 50 per cent.

The effects of notch depth on the fracture stress, Fig. 11, can also be explained by the presence of a considerable stress concentration at the moment of failure in bars provided with notches of intermediate depth. The shape of the fracture stress versus notch depth curves is quite definite for notch ductile conditions. With increasing notch depth, the fracture stress decreases considerably at first, reaching a minimum value of somewhere between 70 and 80 per cent of the fracture stress of the unnotched specimen for a specimen having an area of approximately 10 per cent removed by the notch. With further increasing notch depth, the fracture stress again increases almost linearly and approaches a limiting value somewhere between 300,000 to 350,000 pounds per square inch. As in the case of the notch strength ratio, this upper limit is reached only if the ultimate strength is approximately 200,000 pounds per square inch or less. Steels which are notch brittle because of a higher strength level also show the decrease of fracture stress for shallow notches, but only a slow increase, if any, with further increasing notch depth. For steels which are very strong and notch

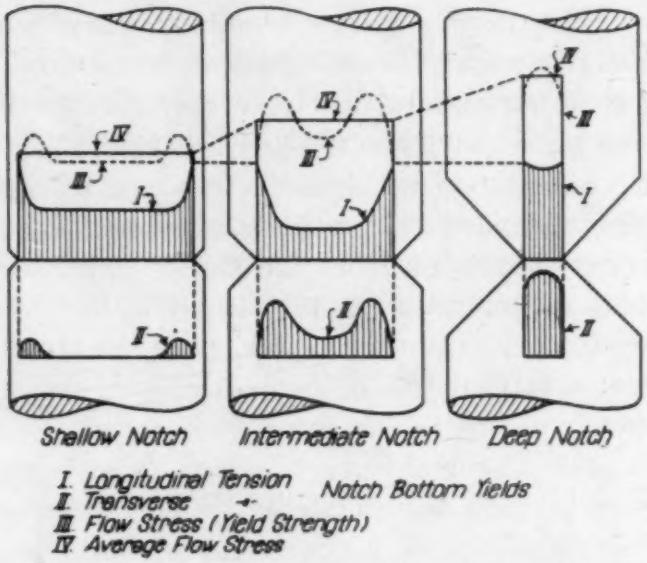


Fig. 15—Diagrammatic Representation of Effect of Notch Depth on the Stress State and the Yield Strength.

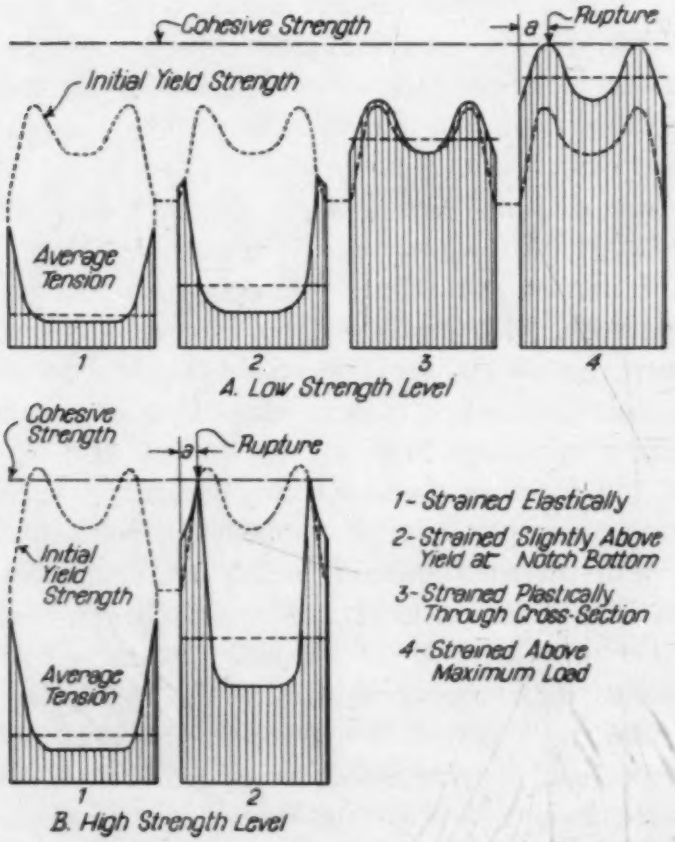


Fig. 16—Diagrammatic Representation of Distribution of Longitudinal Tension Over a Notched Section During Tensile Test.

brittle, the fracture stress of deeply notched specimens was not sufficiently consistent to reveal any relations.

The presence of a minimum in the notch ductility versus notch depth curves, and in the fracture stress versus notch depth curves, agrees with the stress distributions observed in bars having a certain section size and provided with notches of various depths which were subjected to elastic tension. It was found that the stress concentration in such bars is greatest for notch depths between 30 and 40 per cent, with more uniform stress distribution for both larger and smaller notch depths (34).

Stress Distribution and Rupturing—At present, there is no experimental method available which will disclose the stress distribution in a notched cylindrical bar subjected to a tensile test. However, the stress distribution must conform to a number of general laws in both the elastic and the plastic state of stress:

(A) Regarding the stress distribution over the notched section in the elastic state, Fig. 15, the following changes occur on increasing the notch depth:
  (a) The peak stress (of the longitudinal tension) first differs little from the average stress, then increases to a maximum for an intermediate notch depth, and eventually decreases again, approaching the value of the average stress as the notch becomes very deep (44). In other words, the most nonuniform stress distribution is present with notches of intermediate depth.
  (b) The radial transverse stress is always zero at the notch bottom. This also applies to the stress state after plastic flow has occurred.
  (c) The average transverse stress increases with increasing notch depth. The distribution of the transverse stress is characterized by a maximum which is localized and close to the notch bottom for shallow notches, but which becomes flat and close to the center of the bar for deep notches.

(B) Regarding the resistance to plastic flow, or flow stress, Fig. 16, which can be considered as being represented by the yield strength of the steel, the following conclusions can be drawn from the elastic stress distribution:
  (a) At the notch bottom the flow stress is always the same as the yield strength of a cylindrical bar of the same metal.

(b) The flow stress at any point of the notched section is approximately equal to the yield strength of the cylindrical bar plus the transverse stress present at the moment of yielding (1). This results in a distribution of the flow stress, or inherent resistance against plastic flow which reflects the peaks and valleys of the transverse stress.

(C) Regarding the changes of longitudinal stress during plastic flow, a gradual transformation from the elastic state to the state of yielding through the complete section, as represented by the flow stress, should take place. Consequently, plastic flow in a notched bar should begin at the notch bottom and progress gradually towards the center until the complete section is yielding.

(D) The longitudinal stress should increase additionally at any point which is subject to plastic flow because of strain hardening of the steel, Fig. 16.

(E) Rupture should take place when the longitudinal stress at any point of the notched section reaches the technical cohesive strength, Fig. 16. The cohesive strength is less variable than the flow stress and can be considered as constant for the present purpose.

Regarding the condition of rupture, therefore, it can be seen that two different cases must be distinguished. If the total notched section has been strained plastically before failure, the average fracture stress must be greater than the yield strength plus the average transverse stress, which depends mainly upon the notch depth. This includes the ductile and semiductile conditions, the contraction in area being more, say, than 0.2 per cent. This condition will be present if the flow stress at each point of the notched section is initially smaller than the cohesive strength so that the stress reaches the cohesive strength only after some plastic flow and accompanying strain hardening has taken place, Fig. 16. However, if the flow stress, because of a combination of high initial yield strength plus the effect of transverse stress, is higher than the cohesive strength in some ranges of the notched section, failure will occur after the beginning of plastic flow at the notch bottom but before the center of the bar has been plastically strained, Fig. 16. Thus, the stress in the center portion of the bar will stay rather low and a low average fracture stress will result. Exactly how low the fracture stress can be in this case is not clear, as the stress concentra-

tion within a sharply notched bar might be very high initially. This explains the very low values of average fracture stress observed. It is not possible to make a more definite statement at present.

Effects of Tempering Temperature and Hardening Temperature—The strength level of a particular alloy steel is primarily determined by the temperature of tempering, proper quenching being assumed. Therefore, the results of notched bar tensile tests on alloy steels are decidedly dependent upon the tempering treatment applied

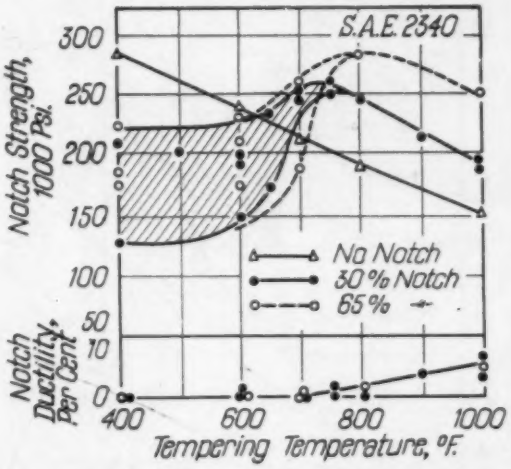


Fig. 17—Effect of Tempering Temperature on Notch Strength and Notch Ductility of a Low Alloy Steel.

after having hardened the steel. This is apparent from the data selected for bars provided with 30 and 65 per cent notches. These data are replotted in Fig. 17 versus the tempering temperature.

The assembly in Fig. 18 of the notch ductility values for the various steels tempered at various temperatures reveals the fundamental fact that the notch ductility of any of the steels investigated decreases progressively with decreasing tempering temperature. When tempered for one-half hour below a temperature of 800 degrees Fahr. (425 degrees Cent.), the notch ductility of all the steels investigated is 1 per cent or less, and within the present limits of accuracy, the steels are completely brittle.

The notch strength, Fig. 17, illustrates the fundamental effect of tempering temperatures, particularly for the temperature range below 800 degrees Fahr. (425 degrees Cent.). At higher temperatures, the notch strength is higher throughout than the ultimate strength of the unnotched specimen by a nearly constant factor, as previously discussed. This condition prevails for tempering tem-

peratures down to approximately 800 degrees Fahr. (425 degrees Cent.), but it is radically changed at this point. The notch strength decreases somewhat at first and at the same time the scattering appears. This scattering becomes very large if the tempering temperature is 600 degrees Fahr. (315 degrees Cent.) or lower, the

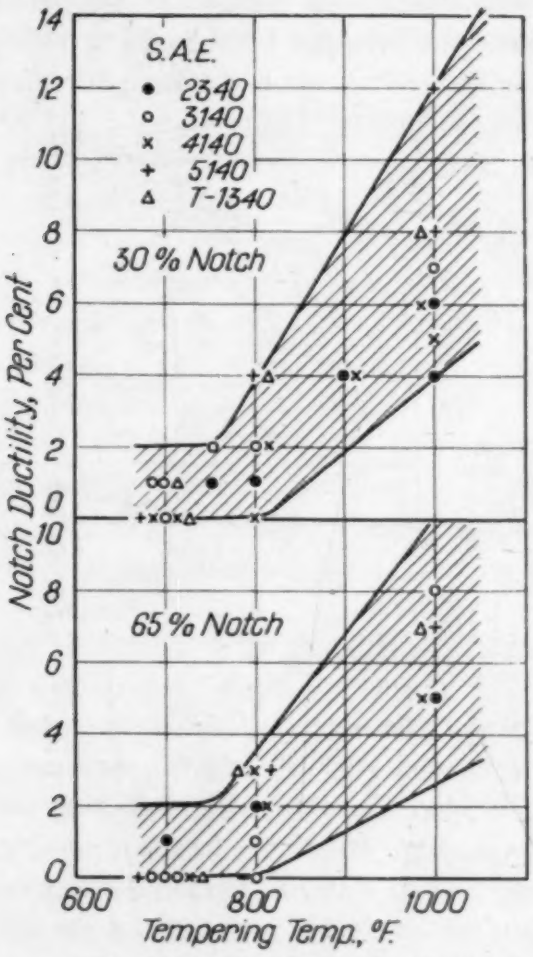


Fig. 18—Effect of Tempering Temperature on the Notch Ductility of the Various Steels.

maximum values being slightly above the ultimate strength. Thus, the notch strength values seem to indicate a radical difference in the notched bar characteristics of the steels, depending on whether they are tempered at temperatures below or above a certain temperature which is approximately 800 degrees Fahr. (425 degrees Cent.).

However, such a conclusion is fundamentally erroneous. Steels showing a contraction in area of, say, 5 per cent, are not considered

as particularly ductile. But this is the average ductility of 30 to 65 per cent notched tensile test bars, quenched and tempered at a temperature of approximately 900 degrees Fahr. (480 degrees Cent.) and having a strength between 170,000 and 200,000 pounds per square inch. Thus, deep notching creates a severe embrittlement in that it reduces the contraction in area of an unnotched specimen from 50 per cent or more to 5 per cent; but this embrittlement is not apparent from the notch strength values.

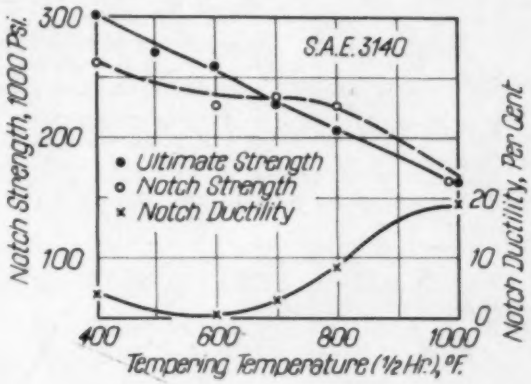


Fig. 19—Effect of Tempering Temperature on Notch Strength and Notch Ductility of 5 Per Cent Notched S.A.E. 3140 Steel Bars Quenched from 1600 Degrees Fahr. and Tempered at Various Temperatures.

This inability of the notch strength to indicate brittleness is a partly mechanical and not a purely metallurgical phenomenon, i.e., the notch strength values are highly dependent upon the particular type of testing. In this connection, it has been previously shown (24), (25), that by additional bending the notch strength might be radically reduced even if the steel is tempered at a temperature as high as 900 degrees Fahr. (480 degrees Cent.), and has an ultimate strength of approximately 160,000 pounds per square inch, while the notch strength of softer and presumably more ductile steels is not affected by additional bending.

The effects of notching on steels having a strength level over 200,000 pounds per square inch are also illustrated by the notch ductility of bars of the S.A.E. 3140 steel provided with a shallow notch, Fig. 19. The ductility of such specimens also decreases with decreasing tempering temperatures but not below some finite, measurable value. A minimum value of notch ductility was reached for any of the steels investigated on tempering at a temperature of

approximately 600 degrees Fahr. (315 degrees Cent.), while tempering at 400 degrees Fahr. yielded somewhat higher values.

Until now, such a relation has not been observed in any static test; but a minimum of "toughness" is ascribed to steels tempered at temperatures close to 600 degrees Fahr. (315 degrees Cent.) on the basis of notched bar impact tests (45). In order to investigate

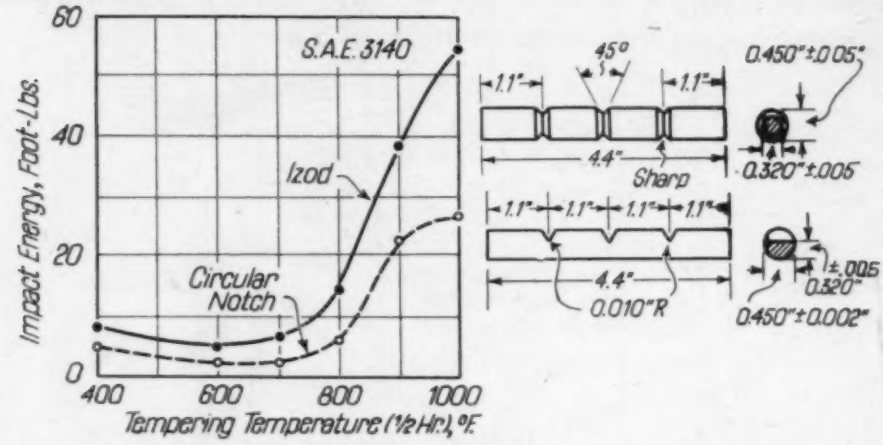


Fig. 20—Results of Two Types of Notched Bar Izod Impact Tests on S.A.E. 3140 Steel.

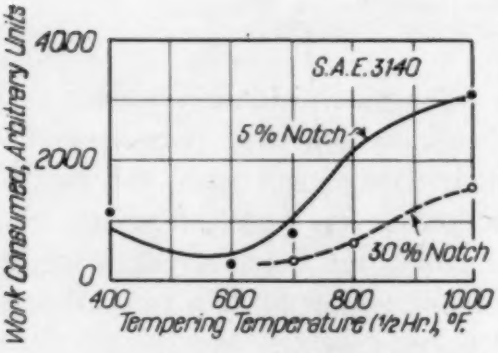


Fig. 21—Work Consumed in Tensile Tests on Notched Bars of S.A.E. 3140 Steel.

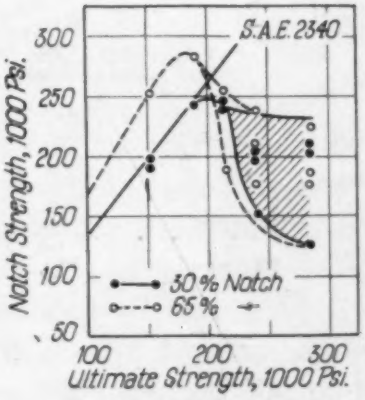


Fig. 22—Relation Between Notch Strength and Strength Level (Ultimate Strength) of a Low Alloy Steel.

such a correlation, a few impact tests were carried out on heat treated bars of the S.A.E. 3140 steel provided with two types of notches. The results of these tests, Fig. 20, show such a minimum for a tempering temperature of about 600 degrees Fahr. (315 degrees Cent.). They also indicate that a sharp circular notch is more severe in impact tests than the more conventional wedge type notch with a fillet.

The values of impact energy might be made comparable to the results of the static tests on notched bars by forming a composite value of the static characteristics which also has the dimensions of energy. This is the product of notch strength and notch ductility, which has been calculated for the S.A.E. 3140 steel bars provided with 5 and 30 per cent notches, Fig. 21. As far as the 30 per cent notched bars yielded reliable values, i.e., for tempering temperatures above 800 degrees Fahr. (425 degrees Cent.), the static, Fig. 21, and the dynamic energy, Fig. 20, depend upon the tempering temperature in much the same manner. The static conditions for low tempering temperatures have been represented only for the 5 per cent notched bars, which also show a similar trend but a slight displacement of the temperature of minimum energy to a lower temperature in comparison with the impact tests. Thus, this comparison shows that the much discussed results of notched bar impact tests are mainly determined by notching and only to a minor extent by the high speed of testing (46).

The progressive embrittlement of a particular steel with increasing strength level can also be evaluated from a representation in which the notch strength of 30 and 65 per cent notched bars has been plotted versus the ultimate strength of the unnotched bars, Fig. 22. Up to an ultimate strength of approximately 200,000 pounds per square inch, all of the steels investigated behaved in practically an identical manner, i.e., their notch strength was a certain multiple of the ultimate strength as presented by the straight lines in Fig. 22. The beginning of embrittlement is indicated by the deviation from this straight line toward lower notch strength values at a certain strength level, followed by the appearance of the large scattering at somewhat higher strength levels.

In order to check the effect of quenching temperature, a few specimens of the S.A.E. 2340 steel were quenched from 1600 and 1750 degrees Fahr. (870 and 955 degrees Cent.). The ultimate strength, elongation, and contraction in area of the unnotched bars were not visibly affected by the quenching temperature, Fig. 23. This fact has been previously observed (24) and cannot be explained as it is well known that the quenching temperature, which determines the austenitic grain size, affects the performance of a steel and its notch impact toughness to a considerable extent. This effect is apparent, however, in the case of the notch strength, Fig. 24, showing that the region of notch brittleness extends further in the direction of higher

tempering temperatures when the hardening temperature is higher. Here again is an indication of the inability of the ordinary tensile properties to reveal the practical performance characteristics of a material, whereas these differences in materials are more readily distinguished by a notched bar tensile test.

Effects of Section Size—A few experiments have been carried

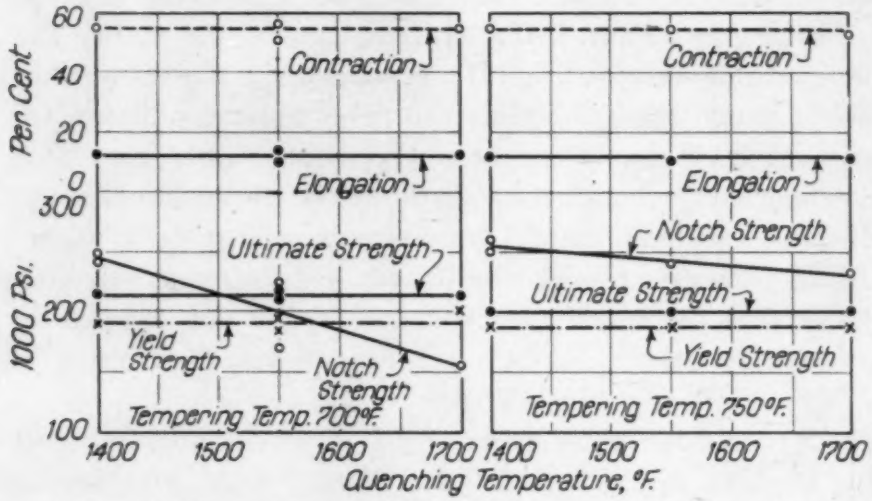


Fig. 23—Effect of Quenching Temperature on Tensile Properties of Heat Treated S.A.E. 2340 Steel.

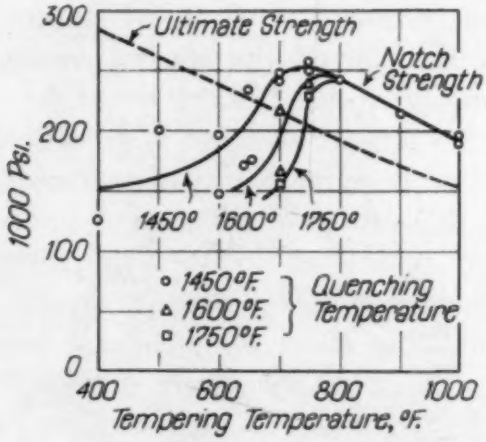


Fig. 24—Effect of Quenching Temperature on Notch Strength of Heat Treated S.A.E. 2340 Steel.

out on the effect of section size. Specimens were made from 2¾-inch diameter hot-rolled rounds with a shape as shown in Fig. 25. These were subjected to the same preliminary and final heat treating procedures as the specimens made from ¾-inch diameter rod, except that for hardening, the specimens were heated for 3 hours in a mix-

ture of powdered charcoal and magnesia. The specimens were designed with a maximum section of ½ inch in order to insure full hardening, thus having 95 per cent of the cross-sectional area removed by notching. The testing fixtures simply consisted of two

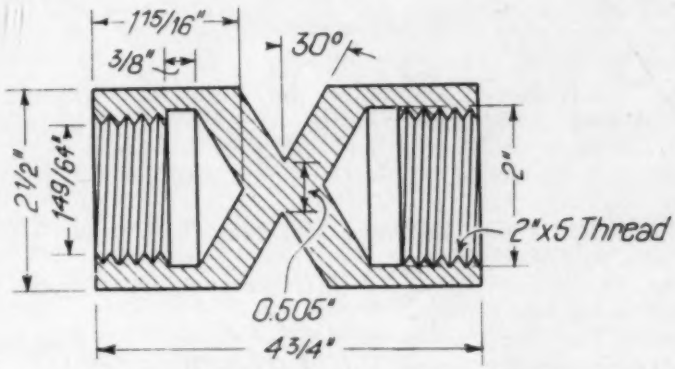


Fig. 25—Large Notched Tensile Test Specimens. Scale One-Third Size.

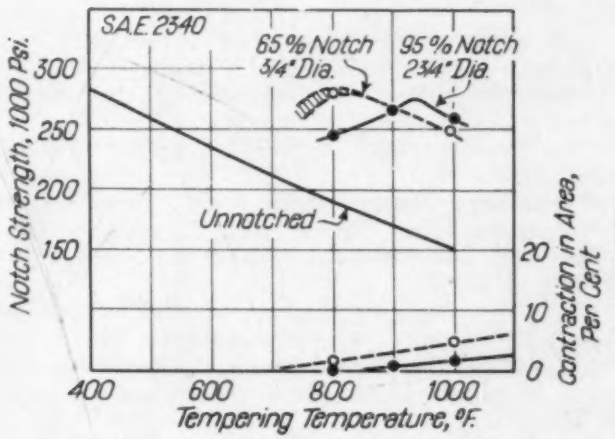


Fig. 26—Effect of Section Size on Notch Strength.

threaded bars, 2 inches in diameter, passing through the heads of the testing machine and held by means of nuts and large washers.

The results are represented in Fig. 26 along with those for the smaller 65 per cent notched bars for comparison.

It can be observed that for a given tempering temperature, the ductility of a large specimen (with a 95 per cent notch) is considerably less than that of a small specimen (with a 65 per cent notch). Consequently, with decreasing temperature, the large bars become brittle, i.e., their contraction in area becomes zero, at a tempering temperature of approximately 150 degrees Fahr. (65 degrees Cent.) higher than that for small sections.

Thus, a large section has a considerably higher tendency towards embrittlement by notching than a small section. This might be explained by the probable relation that a large section generally has a lower inherent ductility (in direction of fiber) than in small section.

Bibliography

1. G. Sachs, "Some Fundamentals of the Flow and Rupture of Metals," *Transactions,* American Institute of Mining and Metallurgical Engineers, Institute of Metals Division, 1941, p. 12-29.
2. P. Ludwik and R. Scheu, "Effect of Notches on Soft Iron," *Stahl und Eisen,* Vol. 43, 1923, p. 999-1001.
3. P. Ludwik, "Yield Point, Cold and Hot Shortness," *Zeit. V. D. Ing.,* Vol. 70, 1926, p. 379-386.
4. W. Kuntze, "Cohesive Strength," *Mitteilungen, D. Materialprüfungsanst.,* Special Issue No. 20, 1932.
5. W. Kuntze, "The Fracture of Notched Tensile Test Specimens," *Archiv für das Eisenhüttenwesen,* Vol. 2, 1928-29, p. 109-117.
6. W. Kuntze, "Impact Strength and Conventional Mechanical Properties," *Archiv für das Eisenhüttenwesen,* Vol. 2, 1928-29, p. 583-590.
7. W. Kuntze, "The Strength of Threads," *Zeit. V. D. Ing.,* Vol. 73, 1929, p. 469-471.
8. W. Kuntze, "The Practical Procedure for Notched Bar Tensile Tests," *Metallwirtschaft,* Vol. 11, 1932, p. 179-184.
9. W. Kuntze, "The Procedure of Determining the Cohesive Strength," *Metallwirtschaft,* Vol. 11, 1932, p. 343-347.
10. W. Kuntze, "The Problems of Cohesion," *Zeit. für Metallkunde,* Vol. 22, 1930, p. 264-268.
11. W. Kuntze, "Transverse Elastic Strain and Triaxial Stress States in Notched Specimens," *Zeit. Physik,* Vol. 72, 1931, p. 785-792.
12. W. Kuntze, "Ductility and Strength of Notched Specimens," *Zeit. Physik,* Vol. 74, 1932, p. 45-65.
13. W. Kuntze, "The Safety Against Rupture of Welded Constructions," *Wiss. Abhandlungen, D. Materialpruefungsanstalten,* No. 2, 1939, p. 11-18.
14. W. Kuntze, "Mechanical Testing of Materials Regarding Their Suitability for Practical Service," *Archiv für das Eisenhüttenwesen,* Vol. 12, 1938-39, p. 329-334.
15. A. Martens, *Zeit. V. D. Ing.,* Vol. 45, 1901, p. 805-812.
16. M. Rudeloff, *Stahl und Eisen,* Vol. 22, 1902, p. 374-380.
17. D. J. McAdam, "The Technical Cohesive Strength of Metals," *Journal of Applied Mechanics,* Vol. 8, 1941, p. A155-165.
18. D. J. McAdam, "The Technical Cohesive Strength and Yield Strength of Metals," *Metals Technology,* January 1942, Tech. Pub. No. 1414.
19. K. Matthaes, "Notch Effects Under Static Stress," *Luftfahrtforschung,* Vol. 14, 1938, p. 28-40; *Metal Treatment,* Vol. 4, 1938, Spring Issue, p. 15-23.
20. N. Davidenkov and H. M. Schewandin, "The Cohesive Strength of Previously Stretched and Compressed Tensile Test Specimens," *Zeit. für Metallkunde,* Vol. 22, 1930, p. 264-268.
21. A. Nadai and C. W. McGregor, "Concerning the Effects of Notches and Laws of Similitude in Material Testing," *Proceedings,* American Society for Testing Materials, Vol. 34, II, 1934, p. 216-228.
22. H. Floessner, "The Mechanical Properties of Notched Bars," Doctor Thesis, Dresden, 1927.
23. N. Davidenkov and F. Wittmann, "Mechanical Analysis of Impact Brittleness," *Technical Physics,* U.S.S.R., Vol. 4, 1937, No. 4, p. 1-17.

24. M. Gensamer, "Static Crack Strength of Metals, Its Determination and Significance," METAL PROGRESS, Vol. 38, 1940, p. 58-64.
25. M. Gensamer, E. B. Pearsall and G. V. Smith, "The Mechanical Properties of the Isothermal Decomposition Products of Austenite," TRANSACTIONS, American Society for Metals, Vol. 28, 1940, p. 380-398.
26. M. Gensamer, E. B. Pearsall, W. S. Pellini and J. R. Low, "The Tensile Properties of Pearlite, Bainite, and Spheroidite," TRANSACTIONS, American Society for Metals, Convention 1941, Preprint No. 19.
27. H. Kiessler and W. Connert, "Notched Bar Tensile Tests with Superposed Bending Strain," *Archiv für das Eisenhüttenwesen,* Vol. 14, 1940-41, p. 555-560.
28. K. Matthaes, "The Notched Bar Impact Test and Its Accompanying Phenomena," Doctor Thesis, Dresden, 1927.
29. G. Sachs, "Stress Raisers," *Iron Age,* Vol. 146, 1940, August 1, p. 31-34; August 8, p. 34-37.
30. E. A. Davis, "Combined Tension-Torsion Tests on 0.35 Per Cent Carbon Steel," *Transactions,* American Society of Mechanical Engineers, Vol. 62, 1940, p. 577-586.
31. H. Neuber, "Exact Elastic Solutions Concerning the Notch Effect on Plates and Bodies of Rotational Symmetry," *Zeit. angew. Mathematik,* Vol. 13, 1933, p. 439-442.
32. E. Preuss, "Experiments Regarding the Stress Distribution in Notched Tensile Test Bars," *Zeit. V. Deut. Ing.,* Vol. 57, 1913, p. 664-667.
33. E. G. Coker and L. G. Eilon, "Photoelasticity," London, 1931, p. 362-597.
34. M. M. Frocht, "Factors of Stress Concentration Photoelastically Determined," *Journal of Applied Mechanics,* Vol. 2, 1935, p. A67-68.
35. A. M. Wahl and R. Beewkes, "Stress Concentration Produced by Holes and Notches," *Transactions,* American Society of Mechanical Engineers, Vol. 56, 1934, p. 617-625.
36. S. Berg, Forschungsarb. Ingenieur Wesen, No. 331, 1930.
37. H. Kraechter, "X-Ray Determination of the Stress Concentration Factor of Notched Cylindrical Test Bars," *Zeit. für Metallkunde,* Vol. 31, 1939, p. 114-115.
38. E. Schmid and W. Boas, "Plasticity of Crystals," Berlin, 1935, p. 176 and 258.
39. R. Ludwik, "The Meaning of Flow and Rupture Resistance in Material Testing," *Zeit. V. D. Ing.,* Vol. 71, 1927, p. 1532-1538.
40. N. Davidenkov and P. Sakharov, "On the Influence of Cold Working Upon the Brittleness of Steel," *Technical Physics,* U. S. S. R., Vol. 5, 1938, p. 743-758.
41. J. E. Docherty, "Slow Bending Tests on Large Notched Bars," *Engineering,* Vol. 139, 1935, p. 211-213.
42. A. F. Maier, "Effect of Stress State on Ductility of Metallic Materials," *Mitt. Materialpruefungs Anst.,* Stuttgart, 1935.
43. E. Siebel and A. Maier, "Effect of Multiaxial Stress States on the Ductility," *Zeit. V. D. Ing.,* Vol. 77, 1933, p. 1345-1349.
44. M. M. Frocht, "The Behavior of a Brittle Material at Failure," *Journal of Applied Mechanics,* Vol. 3, 1936, p. A99-103.
45. Nickel Alloy Steels, Data Sheet No. II, 4.
46. E. J. Janitzky and M. Baeyertz, "The Marked Similarity in Tensile Properties of Several Heat Treated S.A.E. Steels," *Metals Handbook,* 1940.
47. "Notched Bar Testing of Steel," *Metals Handbook,* 1940 Edition, p. 671.
48. S. L. Hoyt, "Notched-Bar Testing and Impact Testing," *Proceedings,* American Society for Testing Materials, Vol. 38, II, 1938, p. 141-156.
49. E. R. Parker and C. Ferguson, "The Effect of Strain Rate Upon the Tensile Impact Strength of Some Metals," TRANSACTIONS, American Society for Metals, Vol. 30, 1942, p. 68-80.
50. E. Siebel, "Static and Dynamic Notch Toughness," *Jahrbuch,* Lilienthal Gesellschaft, 1936, p. 383-396.
51. G. Sachs and J. Lubahn, "Effects of Notching on Strained Metals", *Iron Age,* Vol. 150, 1942, Oct. 8, p. 31-38; Oct. 15, p. 48-52.

DISCUSSION

Written Discussion: By D. J. McAdam, Jr., metallurgist, National Bureau of Standards, Washington, D. C.

In their introductory section, the authors state that they have included a rather complete bibliography with regard to the considerable amount of research by various investigators on the effect of notches. They also state that the interpretation of the results of these investigations is "rather inconclusive and open to argument." As an adequate interpretation of the effects of notches necessarily involves a study of the technical cohesive strength of metals, the authors apparently consider that the studies of this subject in papers listed in the bibliography are inconclusive and open to argument. With the exception of two papers listed, all the authors who discuss the subject of technical cohesion hold the prevalent view that the technical cohesion limit is determined by a value of the algebraically greatest principal stress (tensile stresses being viewed as positive). In the two listed papers by the writer (17, 18), however, evidence is presented that the technical cohesion limit depends on the combination of all three principal stresses. According to this view, the technical cohesive strength of a metal, in any specific condition as regards prior mechanical and heat treatment, cannot be represented by any single stress value, but comprises an infinite number of technical cohesion limits varying with the combination of principal stresses. Diagrams were presented to show qualitatively the influence of the combination of principal stresses and of plastic deformation on the technical cohesion limit.

The evidence presented in the two papers by the writer was derived from published results of tension tests (by other investigators) of notched specimens. The results confirm the results of compression tests of various kinds by Bridgman. About 30 years ago, Bridgman published the first of a series of papers with evidence and conclusion that the technical cohesion limit varies greatly with the combination of principal stresses. Although Bridgman's experiments were compressive tests on unnotched specimens, they should be considered in connection with the bibliography given by the authors. For this purpose, they are listed below.[9] The combined evidence presented in these papers and the two papers by the writer (17, 18) should lead to the long overdue abandonment of the view that fracture is determined by a limiting value of the algebraically greatest principal stress.

The authors apparently are not yet ready to abandon this theory. In Fig. 16, for example, the technical cohesive strength is represented by a horizontal line (which may be viewed as the intercept of a horizontal plane), thus implying that the technical cohesion limit is unaffected by the variation of the radial stress from zero at the circumference to a maximum somewhere in the interior. Actually, however, the cohesion limit varies greatly with the ratio of the radial to the longitudinal stress. It also varies greatly with the varying plastic deformation existing in the locally contracted section at fracture. The location

[9] P. W. Bridgman, "Breaking Tests Under Hydrostatic Pressure, and Conditions of Rupture," *Philosophical Magazine*, July 1912, Vol. 24, p. 63-80; "Theoretically Interesting Aspects of High-Pressure Phenomena," *Reviews of Modern Physics*, 1935, Vol. 7, p. 1-35; "Reflections on Rupture," *Journal Applied Physics*, 1938, Vol. 9, p. 517-528; "Considerations on Rupture Under Triaxial Stress," *Mechanical Engineering*, Feb. 1939, Vol. 61, p. 107-111.

of the origin of fracture depends on the relation between the variations of radial stress and plastic deformation, as well as on the variation of longitudinal stress.

The writer can find nothing in this paper that is not in accordance with the idea that the technical cohesion limit varies with the combination of the principal stresses and finds considerable evidence in support of this idea. In Fig. 11, for example, the diagram for steel tempered at 1000 degrees Fahr. (540 degrees Cent.) can be explained readily in terms of the views presented in papers by the writer (17, 18). The authors, however, probably would interpret the curves in terms of variation of stress concentration. In the only specific reference to the writer's papers, on page 145, the authors incorrectly attribute to the writer the idea that stress concentration "is eliminated by a plastic strain of approximately 0.5 per cent". On page 3 of the second of the two papers (18) is the statement: "A *large part* of the stress concentration, therefore, evidently is removed by the 0.6 per cent plastic deformation." It was emphasized, moreover, that any stress concentration remaining at either maximum load or fracture would not be sufficient to vitiate the conclusions expressed in the two papers. Since those two papers were published, the writer has given much thought to the subject and finds no reason to change his views that the diagrams there presented are qualitatively correct.

The writer cannot agree with the idea expressed on pages 137 and 138 that the notch ductility is "that value obtainable in the notched bar tensile tests which is subject to the most fundamental generalizations." The reduction of area observed after fracture is an unsatisfactory measure of the plastic deformation, and the value measured probably is the least "fundamental" of the values obtained in the tension test of a notched specimen.

The fact that a nearly constant "notch strength ratio" (Fig. 8) was obtained for the same notch when the notch ductility is more than about 5 per cent is merely a manifestation of the fact that the same notch caused practically the same ratio of radial to longitudinal stress for all the steels tested. For the steels with notch ductility greater than about 5 per cent, the ultimate stress ("notch strength value") was only a few per cent greater than the yield stress, a percentage of the same order as the range of vertical scatter at abscissas beyond 5 per cent in Fig. 8. The same notch thus caused about the same percentage elevation of the yield stress for all the steels used. This relationship could have been predicted.

Oral Discussion

DANIEL ROSENTHAL:[10] I have an after-thought. I should like to ask Professor Sachs what would be the result of his tests if the notched specimen prior to testing were subjected to a plastic compression. So far as I remember there is an indication in the literature that this will deteriorate the behavior of the notched specimen. If this is true then there might be perhaps an explanation for the relation of the plastic deformation and the resistance of notched specimens.

V. N. KRIVOBOK:[11] Since this discussion is more or less in the nature of

[10]Assistant professor of metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

[11]Chief metallurgist, Lockheed Aircraft Corp., Burbank, Cal.

asking questions of our eminent friend, Dr. Sachs, I should like to ask a question too, if I may be allowed.

What is the effect of the rate at which the stress was applied on the results of notched bar testing? Fundamentally, it is an important question because I find that we are rather negligent in discussing that range of S-N curve which lies between the value of tensile strength and the values for fatigue life.

Oftentimes this range is referred to as part of the fatigue testing, yet it should be more properly called repeated impact. What is the effect of the rate of test on the distribution of stress under this latter condition of testing is indeed a moot question. It would indeed be most interesting if Dr. Sachs would throw some light on this inquiry.

Authors' Reply

We have studied Dr. McAdams' interesting work, but it appears rather difficult to answer his discussion remarks. He advances the theory that the condition of failure in any metal primarily subjected to tension is not a constant value of tension stress but depends upon the presence of tension in other directions. We do not either disagree or agree with his theory, but consider the experimental evidence so far obtained as not sufficient to support any advanced theory of failure. At present, no method is apparent to us which permits the determination of the actual stresses (rather than average stresses) in notched tensile test bars. Consequently, we have used the simplest possible conception to develop relations which do not depend materially upon any particular theory of failure.

The notch ductility, or average transverse ductility, is not being considered by the authors to be a simple function, but it was found surprisingly to be the decisive factor regarding the effect of notching on the strength, producing relations of much the same type as between the normal ductility and the ultimate strength of the metal. The finding that the notch strength ratio is almost constant if the notch ductility is high (above 5 per cent) is explained by us in the same manner as by Dr. McAdam. However, this does not apply exactly to the yield strength, which is quite low in sharply notched bars, at least if it is defined for rather small strains. Thus, we do not quite agree with the statement that the results could have been predicted.

Answering the question of Professor Rosenthal, it has been previously observed (10), (20), (51) that prestressing in tension considerably raises the notch strength ratio, while prestressing in compression considerably lowers the notch strength ratio. This effect much resembles that of straining in compression and tension, respectively, on the yield strength in tension ("Bauschinger effect") and consequently may be explained as caused by residual stresses of short range.

Answering the question of Dr. Krivobok, we have not carried out notched bar tensile tests at high speeds. We have pointed out, however, in discussing previous literature (51) that the embrittling effect of notching is considerably greater than that of increased speed, confirming Hoyt's conceptions on this subject (48).

THE KINETICS OF AUSTENITE DECOMPOSITION IN HIGH SPEED STEEL

By Paul Gordon, Morris Cohen and Robert S. Rose

Abstract

The austenite decomposition in 6-6-2 and 18-4-1 high speed steels was studied by means of hot quenching experiments in the range of 210 to 1400 degrees Fahr. (100 to 760 degrees Cent.). With the aid of a suitable dilatometer, it was possible to observe the transformations during cooling to, holding at, and cooling from the hot quenching temperatures. A quantitative metallographic technique was employed to convert the dilation measurements to per cent transformation on an absolute basis. This procedure permitted the construction of detailed transformation curves (commonly called S-curves).

The transformation curves for both steels are very much alike, and consist of two families of C-curves with the lower family intercepted by a set of almost horizontal lines. In the upper transformation range, the product is pearlite, preceded or accompanied by carbide precipitation. The lower C-curves represent the decomposition of austenite into bainite. Bainite formation has a stabilizing influence on the remaining austenite, since the transformation does not go to completion and the M-point of the remaining austenite is depressed, in some cases even below room temperature.

On hot quenching into the martensite range, martensite forms during the cooling to the holding temperature. After the holding temperature is reached, a two-stage isothermal transformation takes place. The first stage proceeds slowly with the appearance of bainite plates in the microstructural regions occupied by the martensite. This transformation is small in extent, but is sufficient to cause a slight upward slant of the otherwise horizontal lines in the martensite range. The second stage of isothermal transformation results in the general formation of bainite throughout the structure, and occurs at the time indicated by the bainite C-curves extended down from the higher temperatures.

A paper presented before the Twenty-fourth Annual Convention of the Society held in Cleveland, October 12 to 16, 1942. Of the authors, Paul Gordon is research assistant and Morris Cohen is associate professor of physical metallurgy, Massachusetts Institute of Technology, Cambridge, Mass., and Robert S. Rose is sales metallurgist, Vanadium-Alloys Steel Co., Boston. Manuscript received July 16, 1942.

Austenite decomposition does not take place on hot quenching into the bay between the two families of C-curves, even if the holding time is prolonged for 165 hours. However, at these temperatures, a conditioning reaction occurs which facilitates the formation of bainite at the expense of martensite during the subsequent cooling to room temperature. Such bainite formation, unlike the martensite, can be suppressed by rapid cooling from the hot quenching temperature. Controlled combinations of martensite, bainite and retained austenite may be secured by suitable hot quenching treatments, but the practical usefulness of these structures has yet to be determined.

The results of the present investigation have a direct bearing on the common practice of hot quenching high speed steel tools as a part of the hardening operation, and certain precautions are indicated.

Introduction

THE study of isothermal transformations by hot quenching methods has proved so fruitful in the case of plain carbon (1)[1] and intermediate alloy steels (2), (3) that it is now logical to apply such techniques to the higher alloy steels. This is particularly true for the high speed steels since any possible improvements which can be achieved in these vital materials by isothermal treatments would be of positive industrial importance. At the same time, the hot quenching of high speed steel seems to offer an attractive field for scientific investigation inasmuch as the well-known sluggishness of this type of steel should permit more detailed observations than are practicable with the more reactive steels. Therefore, it is quite conceivable that the kinetic behavior of the austenite decomposition in high speed steel, even though complicated, may be more amenable to unique interpretation than much of the work based on the simpler steels.

It is common practice to hot quench high speed steel, not for attaining isothermal transformations, but rather as a means of avoiding difficult cleaning operations and preventing breakage during the hardening. This procedure involves quenching from the hardening temperature into a liquid bath at a temperature designed to suppress the formation of pearlite, and thus enable the steel to harden on leisurely cooling to room temperature. However, despite the fre-

[1]The figures appearing in parentheses refer to the bibliography appended to this paper.

quent usage of this practice, the real effects of the liquid bath treatments are not well understood, and accordingly, there is no general agreement on the proper time-temperature conditions for such hot quenching.

In the present investigation, it is proposed to study by means of dilatometric and metallographic measurements the austenite decomposition in 6-6-2 and 18-4-1 high speed steels during a systematic series of hot quenching treatments and also during the subsequent cooling to room temperature. Particular attention will be paid to the transformations occurring within the martensite range in an attempt to disentangle the cooling and isothermal decompositions which have caused so much confusion in the plain carbon and low alloy steels. This paper is concerned principally with the kinetics and products of the primary austenite transformations. The effect of tempering after hot quenching and the resulting mechanical properties will be covered in future publications.

Previous Work

The literature on the hot quenching of high speed steel, although relatively meager, indicates some advantage for this type of treatment over direct cooling to room temperature. Bornatsky (4) has reported an increase of 90 per cent in the life of 18-4-1 lathe tools as a result of hot quenching to 1200 degrees Fahr. (650 degrees Cent.) for 3 hours instead of oil quenching directly to room temperature. Along the same lines, McCarthy (5) found that hot quenching improved the impact strength of 18-4-1 high speed steel, and concluded that hot quenching to 1050 to 1150 degrees Fahr. (565 to 620 degrees Cent.) produced better lathe tools. DeLong and Palmer (6) observed many years ago that hot quenching in the range of 700 to 1300 degrees Fahr. (370 to 705 degrees Cent.) was favorable for straightening high speed steel tools before the onset of the hardening transformations during the subsequent cooling to room temperature.

From a kinetic point of view, DeLong and Palmer (6) noted that the austenite in high speed steel is very slow to transform when supercooled to 1100 degrees Fahr. (595 degrees Cent.), and that such hot quenching could not be employed to achieve hardening and tempering in the same operation. More recently, Ham, Parke and

Herzig (7) determined qualitative S-curves for 6-6-2 high speed steel. Using dilatometric, hardness, and metallographic tests, these investigators showed three temperature ranges of isothermal activity: 1500 to 1250 degrees Fahr. (815 to 675 degrees Cent.), 600 to 500 degrees Fahr. (315 to 260 degrees Cent.), and 300 degrees Fahr. (150 degrees Cent.) to room temperature, the latter range suggesting isothermal formation of martensite and a lowering of the M-point with increased quenching velocities. From 1100 to 700 degrees Fahr. (595 to 370 degrees Cent.), no decomposition of the supercooled austenite was observed within 11½ days. Qualitative S-curves for 18-4-1 steel have been published by Payson (7A). The kinetics of the austenite transformation in 18-4-1 and several low alloy molybdenum high speed steels were studied by Gulyaev (8) and by Steinberg and Zyuzin (9). They found that (a) there are two temperature ranges of rapid isothermal decomposition of the austenite: 1400 to 1200 degrees Fahr. (760 to 650 degrees Cent.), and 750 to 400 degrees Fahr. (400 to 205 degrees Cent.); (b) the austenite decomposition in the lower range is accompanied by the formation of "martensite-like" needles, but does not go to completion as was also shown by Ham et al. (7); (c) this decomposition lowers the temperature of the austenite-martensite transformation during subsequent cooling; and (d) the isothermal decomposition in the lower temperature range raises the M-point of the retained austenite after tempering.

McCarthy (5) also investigated the cooling transformations following 1-hour hot quenches to various temperatures. Using a magnetic-change method, he observed that the austenite transforms in three stages during cooling from hot quenching temperatures above 975 degrees Fahr. (525 degrees Cent.), but only in one stage if the hot quenching temperature is below 975 degrees Fahr. (525 degrees Cent.). Meier (10), however, found two stages of transformation after hot quenching at 1100 degrees Fahr. (595 degrees Cent.).

It is evident that the present literature does not provide a complete or consistent picture of the kinetics of austenite decomposition during the hot quenching of high speed steel. Further studies on this subject are needed for practical as well as scientific reasons.

Experimental Details

Steels Investigated—The chemical analyses of the two steels

studied in this investigation are given in Table I. The steels will hereafter be referred to as 6-6-2 and 18-4-1, both of which are standard commercial grades of high speed steel. All of this stock was obtained in the form of ¼-inch diameter centerless-ground rods.

Table I
Composition of High Speed Steels

Designation	C	Mn	Si	S	P	W	Cr	V	Mo
6-6-2	0.81	0.24	0.26	0.007	0.016	5.95	4.10	1.64	4.69
18-4-1	0.73	0.21	0.33	0.006	0.015	17.80	4.39	1.09	0.18

Because of the present restriction on the usage of 18-4-1 and the growing importance of 6-6-2 as a substitute, the emphasis of the present work was placed on the latter grade. The results reported here for the 6-6-2 steel are closely applicable to the W.P.B. Type III tungsten-molybdenum high speed steels containing 0.75 to 0.90 per cent carbon, 5.00 to 6.25 per cent tungsten, 4.00 to 6.00 per cent molybdenum, 3.50 to 5.00 per cent chromium, and 1.25 to 2.00 per cent vanadium.

Dilatometric Technique—The first hot quenching runs were made with an Oesterle-type dilatometer (11) because it permitted a rapid transfer of the specimen in the dilatometer from the austenitizing to the hot quenching temperature. However, due to contact with traces of iron oxide from the specimens at the high heat temperatures, the quartz parts of the dilatometer deteriorated quickly, and frequent repairs were necessary. Consequently, a new dilatometer was designed to allow the specimens to be heated separately for austenitizing and then be rapidly inserted in the dilatometer for the hot quenching operation. Moreover, it was deemed essential in this work that the changes in length be ascertained with respect to temperature as well as time, and so a thermocouple in contact with the specimen had to be provided.

The details of the dilatometer are shown schematically in Fig. 1. It consisted of an outer quartz tube ½-inch o.d. by 15 inches long, an inner quartz tube ¼-inch o.d. by 14 inches long, and a third quartz tube $\frac{1}{16}$-inch o.d. by 13 inches long within the ¼-inch tube. An Ames dial gage, readable to ± 0.00001 inch, was fastened rigidly to the outer tube by a method fully described elsewhere (12). The bottom of the outer tube was sealed with an inverted dome, and a 3-inch slot-like opening provided in the lower part of the tube for

admitting the specimen, which then rested on the dome-shaped bottom and transmitted its changes in length through the ¼-inch tube to the dial gage. The innermost tube served merely to insulate one of the chromel-alumel thermocouple leads which entered the dilatometer through the upper part of the ¼-inch tube. The hot junction of the thermocouple projected about ¾ inch below the ¼-inch tube.

The dilatometer specimens were ¼ inch in diameter with the

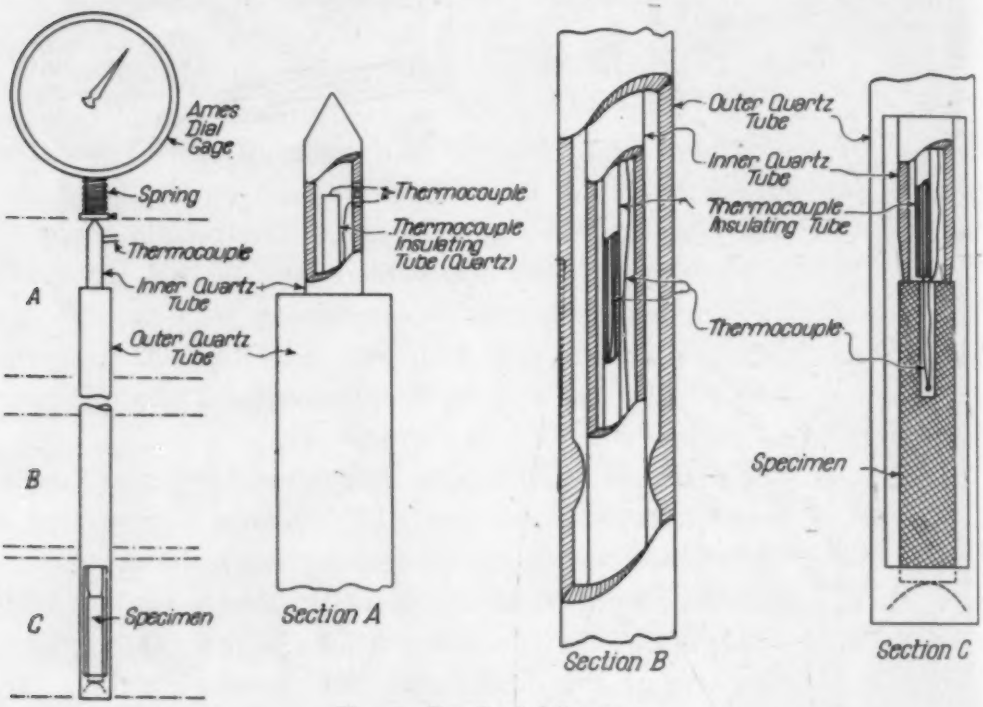


Fig. 1—Detail of Dilatometer.

ends squared off to a length of 2.00 ± 0.01 inches. A $\frac{1}{16}$-inch thermocouple hole was drilled to a depth of ¾ inch along the axis of each specimen. It took less than 45 seconds to lower the hot quenching furnace away from the empty dilatometer, transfer the specimen from the high heat furnace to the dilatometer, raise the intermediate tube so that the thermocouple junction could be slipped into the specimen hole, straighten the specimen so that it rested firmly on the dome-shaped bottom and made good contact with the intermediate tube, clamp the dial gage in position, and finally raise the hot quenching furnace to its former position around the dilatometer. Considering the inherent sluggishness of the steels under investigation, this procedure was highly satisfactory.

The high heat treatments were carried out in a globar furnace provided with a carbonaceous muffle to minimize scaling and decar-

burization. Hardening temperatures of 2235 ± 5 degrees Fahr. (1225 ± 3 degrees Cent.) for the 6-6-2 steel and 2350 ± 5 degrees Fahr. (1290 ± 3 degrees Cent.) for the 18-4-1 were used without preheating. The hot quenching temperatures ranged from 1400 to 210 degrees Fahr. (760 to 100 degrees Cent.). Successful hot quenching experiments could be made in a molten salt or a lead alloy bath, but the solidification of these liquids on the specimen and dilatometer during the cooling after the hot quench prevented an accurate study of the cooling transformations. Since the latter transformations proved to be fully as significant as the isothermal changes themselves, it became necessary to try hot quenching into an air furnace. This procedure gave excellent results provided that the time at the isothermal temperature was corrected to take into account the slower rate of cooling between the austenitizing and hot quenching temperatures. Fortunately, it was discovered that the time for the beginning of isothermal transformation at a given temperature as determined by liquid bath quenching was substantially the same as the sum of the time required by an air-quenched specimen to cool from the eutectoid temperature to the holding temperature plus the isothermal time thereafter to the beginning of transformation. In other words, the effective isothermal time at any given holding temperature attained by air quenching could be obtained by adding to the observed isothermal time the period elapsed during the cooling from the eutectoid to the holding temperature. This method of establishing the effective time at temperature for hot quenching in the air furnace made it possible to use this technique whenever it was desirable to follow the cooling transformations to room temperature after the hot quenching treatment. No claim is held for the scientific rigor of this method of correcting the isothermal time since undoubtedly the procedure would not be suitable for the more reactive steels. With the high speed steels, however, the correction was usually small compared to the actual isothermal times involved.

A photograph of the dilatometer and air quenching furnace is shown in Fig. 2. The method of raising and lowering the furnace is evident. Temperature control of ± 3 degrees Fahr. over a 4-inch zone was obtained. Changes in length due to temperature fluctuations within this range were cancelled before plotting by means of the known temperature coefficients of expansion. In order to control the rate of cooling to room temperature after the isothermal

treatment, the furnace was provided with air jets and a copper cooling coil. Except in the experiments designed to study the effect of cooling rate after hot quenching, a standard rate of 5 to 10 degrees Fahr. per minute was adopted, and the specimens were held in the

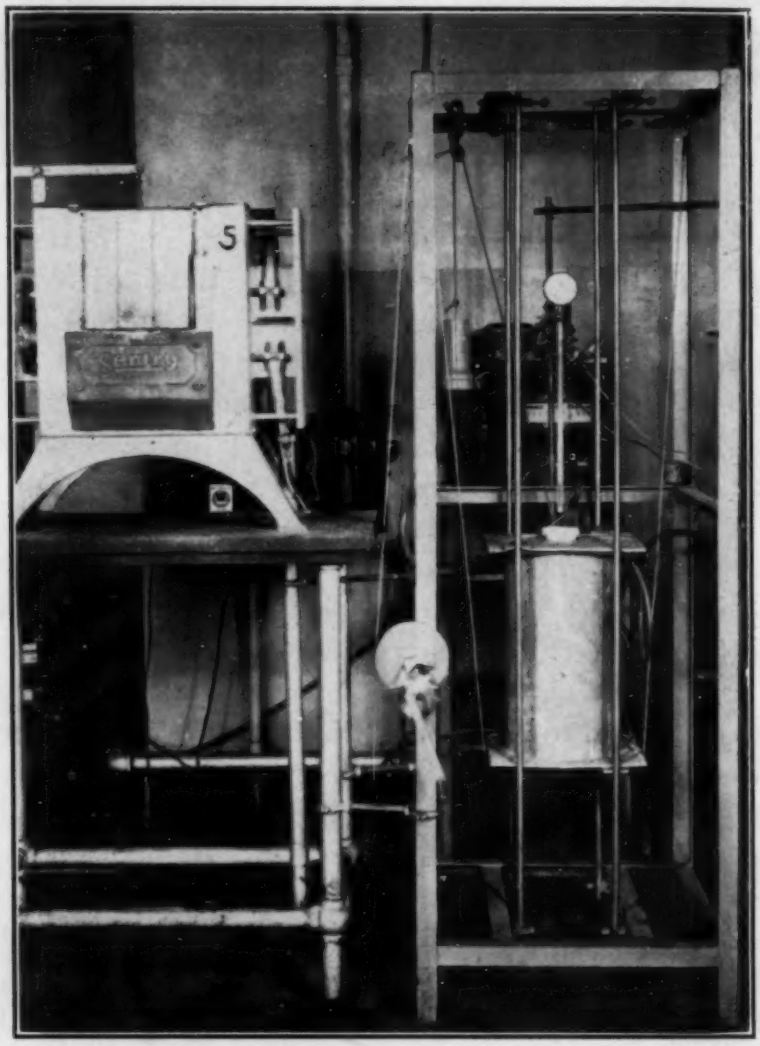


Fig. 2—General View of the Dilatometer, Air Quenching Furnace, and Hardening Furnace.

dilatometer for 24 hours after reaching room temperature. In some of the runs, the cooling was continued to —310 degrees Fahr. (—190 degrees Cent.) with the aid of liquid nitrogen.

Metallographic Technique—Metallographic observations were made (a) to check the beginning of isothermal transformation as indicated by the dilatometer, (b) to study the nature of the various transformation products, (c) to calibrate the dilatometer measure-

ments so that the latter could be translated into per cent transformation on an absolute basis, and (d) to supplement the dilatometer measurements within the martensitic range.

(a) The beginnings of isothermal transformation as determined microscopically on small specimens 0.20 inch in diameter by 0.01 inch thick by the Davenport-Bain hot quenching technique (1) above the M-point and by the Greninger-Troiano double hot quenching technique (13) below the M-point were in excellent agreement with the dilatometric results, and completely justified the previously discussed method of correcting for the slower cooling time of the air-quenched dilatometer specimens. In order to obtain rapid quenching, the customary liquid baths were used for these metallographic experiments. In the double hot quenching procedure, the second hot quench invariably consisted of a 5-second immersion in molten lead at 1050 degrees Fahr. (565 degrees Cent.) in order to darken the transformation products formed during cooling to, and holding at, the first hot quenching temperature.

(b) By ordinary metallographic means, it was found possible to establish carbides, fine pearlite, bainite and martensite as transformation products in both steels. Twelve per cent nital and Murakami's reagent were used for etching.

(c) In order to calibrate the dilatometric measurements, it was necessary to determine quantitatively the per cent of isothermal transformation during certain dilatometer runs. This was done by tempering the isothermally treated specimens for 5 seconds at 1050 degrees Fahr. (565 degrees Cent.), as in the Greninger-Troiano technique (13), in order to darken the transformation product without affecting the remaining austenite. This austenite or its transformation product formed during cooling from 1050 degrees Fahr. (565 degrees Cent.) remained white, and furnished a contrasting background for the dark-etching isothermal product. Ten evenly spaced photomicrographs were taken along a diameter on transverse sections cut through the dilatometer specimens. The photographic conditions were adjusted to secure high contrast in the final prints. Then, by placing translucent graph paper over the prints mounted on a viewing screen, and counting the number of line intersections on the graph paper falling over the black transformation products, the number falling over the white matrix, and the number falling over the white undissolved carbides, the volume percentage of each

constituent could be calculated for each print. By counting 1500 points on 3 by 4-inch prints, independent observers were able to check each other well within ± 2 per cent. Magnifications were selected in accordance with the structure to give clear resolution over as large an area as possible. By averaging the results for the prints corresponding to each specimen, the per cent of isothermal transformation during the dilatometer run was obtained and compared with the isothermal dilation that had been observed for the specimen. The application of this calibration method to the determination of quantitative transformation curves will be discussed later. All of the specimens contained about 5 per cent of undissolved carbides by volume.

(d) While the martensite formation could be readily followed by the dilatometer during the hot quenching to temperatures below the M-point, the rates of cooling attainable were not sufficiently great to permit definite conclusions as to the possible suppressibility of the M-point by drastic quenching, and as to the shape of the lower part of the transformation curves. Consequently, the Greninger-Troiano technique was applied to small specimens quenched to different temperatures within the martensite range for 10 seconds. The martensite thus formed and darkened by a 5-second temper at 1050 degrees Fahr. (565 degrees Cent.) was measured quantitatively by the previously described point-counting procedure. Good agreement with the dilatometric method was found, thus signifying that more rapid cooling than was attainable in the dilatometer would not have measurably altered the per cent transformations within the martensitic range.

Experimental Results

Effect of Hot Quenching Temperature—Figs. 3, 4, 5 and 6 present the dilatometer results of hot quenching the 6-6-2 and 18-4-1 steels to a series of holding temperatures for 24 hours (except for the 1400 degrees Fahr. runs), and then cooling to room temperature at a rate of 5 to 10 degrees Fahr. per minute. Each curve depicts the changes in length undergone by a specimen during cooling to, holding at, and cooling from the hot quenching temperature. For comparison purposes, the dilation curve of a specimen directly cooled to room temperature from the high heat treatment is drawn in dotted lines on each set of curves. The insert at the right of each figure

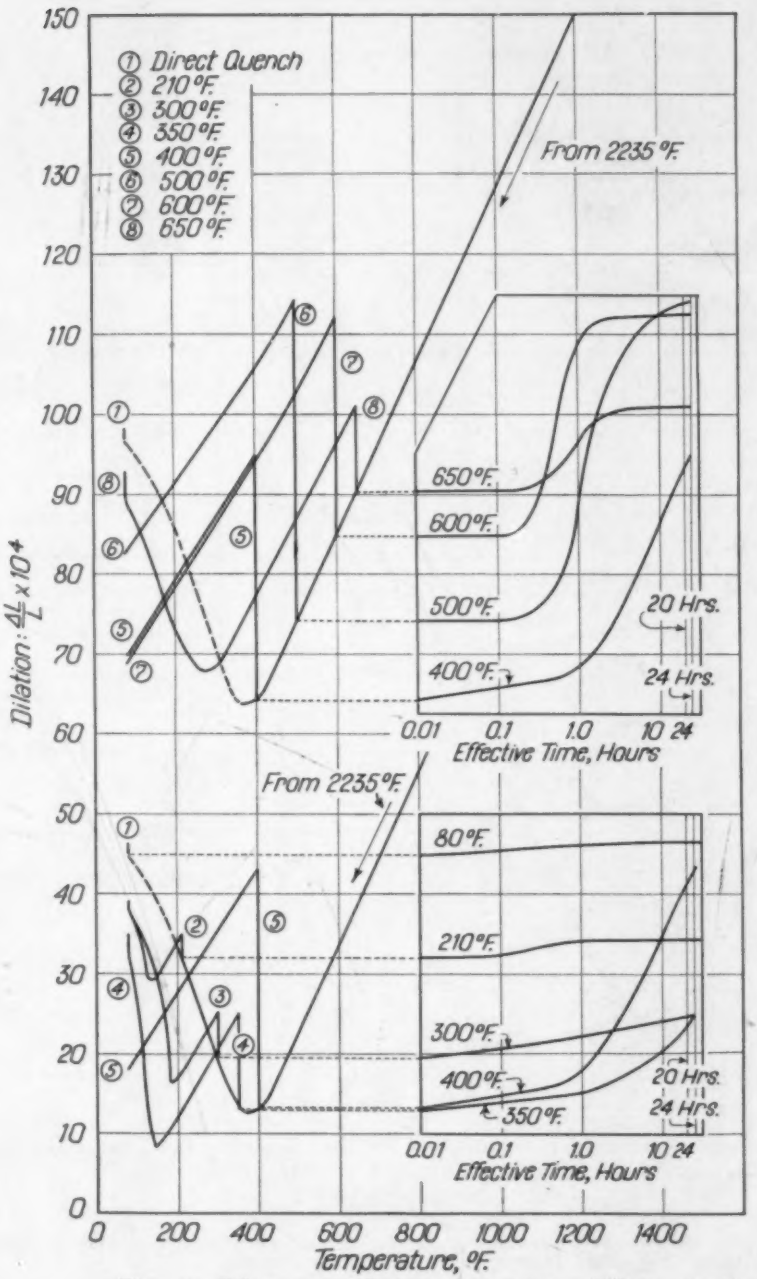


Fig. 3—Dilatometer Curves for 6-6-2. Effect of Hot Quenching Temperature. Holding Time—24 Hours.

shows the isothermal transformation as a function of effective time. It might be helpful to trace out one dilatometer run to demonstrate the way in which these diagrams are read. Consider curve 8 in Fig. 3 representing the hot quenching of 6-6-2 at 650 degrees Fahr. (345 degrees Cent.). From the high heat temperature of 2235 to 650 degrees Fahr. (1225 to 345 degrees Cent.), the specimen con-

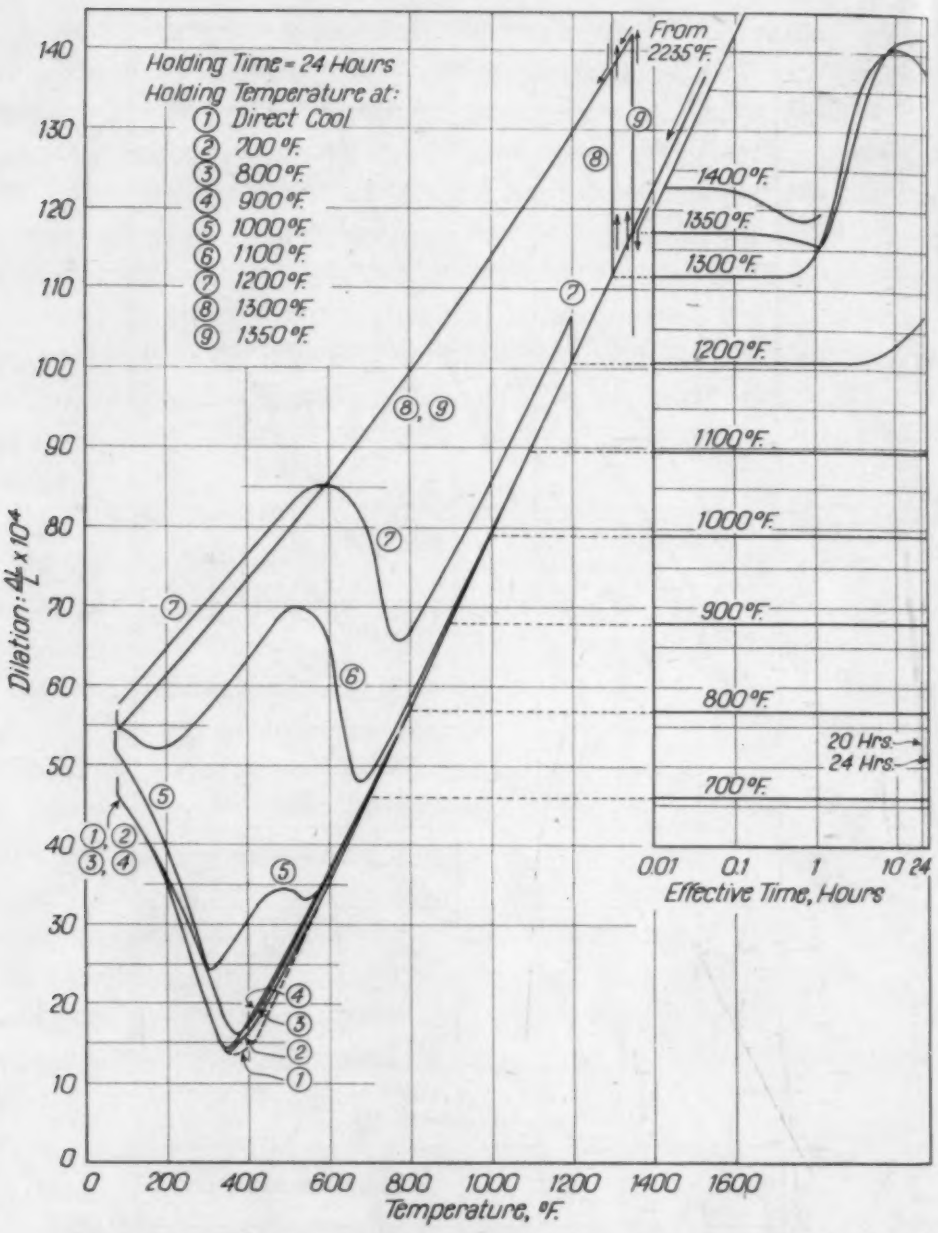


Fig. 4—Dilatometer Curves for 6-6-2. Effect of Hot Quenching Temperature. Holding Time—24 Hours.

tracts normally according to the thermal coefficient of high speed steel in the austenitic condition. On holding at 650 degrees Fahr., the specimen expands 10.5 x 10^{-4} inches per inch in 24 hours. A glance at the 650 degrees Fahr. (345 degrees Cent.) curve in the insert to the right indicates that this isothermal expansion sets in after an incubation period of about 0.18 hour, progresses at an increasing, then decreasing rate, and finally stops well within 24 hours.

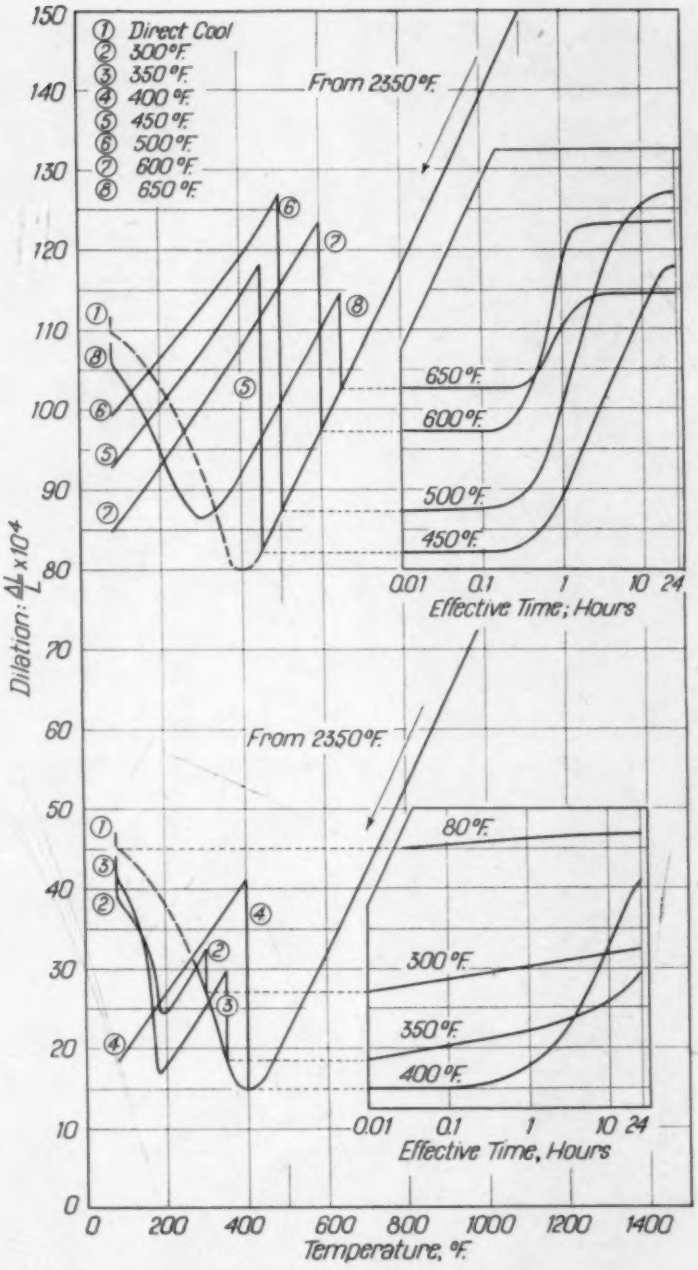


Fig. 5—Dilatometer Curves for 18-4-1. Effect of Hot Quenching Temperature. Holding Time—24 Hours.

However, the austenite transformation is not complete in this period because (returning to the cooling curve 8 on the left) during the subsequent cooling the steel contracts normally only down to 320 degrees Fahr. (160 degrees Cent.) where a marked change in slope appears, indicating the start of a transformation which continues to room temperature. There is also some isothermal transformation at room temperature.

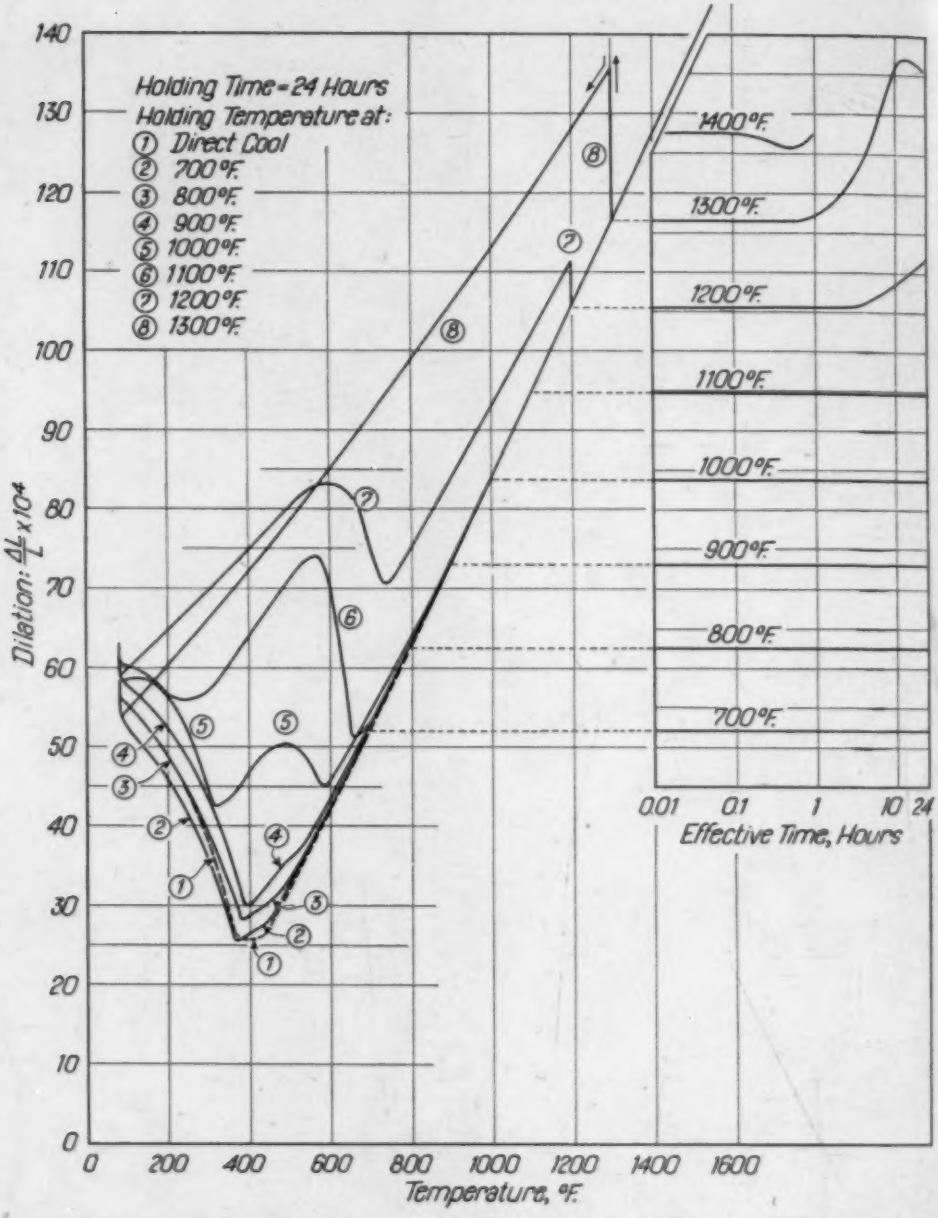


Fig. 6—Dilatometer Curves for 18-4-1. Effect of Hot Quenching Temperature. Holding Time—24 Hours.

The curves in Figs. 3 to 6 and the photomicrographs in Figs. 7 to 8 reveal the following facts about the hot quenching of 6-6-2 and 18-4-1 high speed steels:

1. The hot quenching phenomena are very much alike in both steels.

2. There are two ranges of isothermal activity, one at 1400 to 1200 degrees Fahr. (760 to 650 degrees Cent.) and the other at 675 to 350 degrees Fahr. (355 to 175 degrees Cent.).

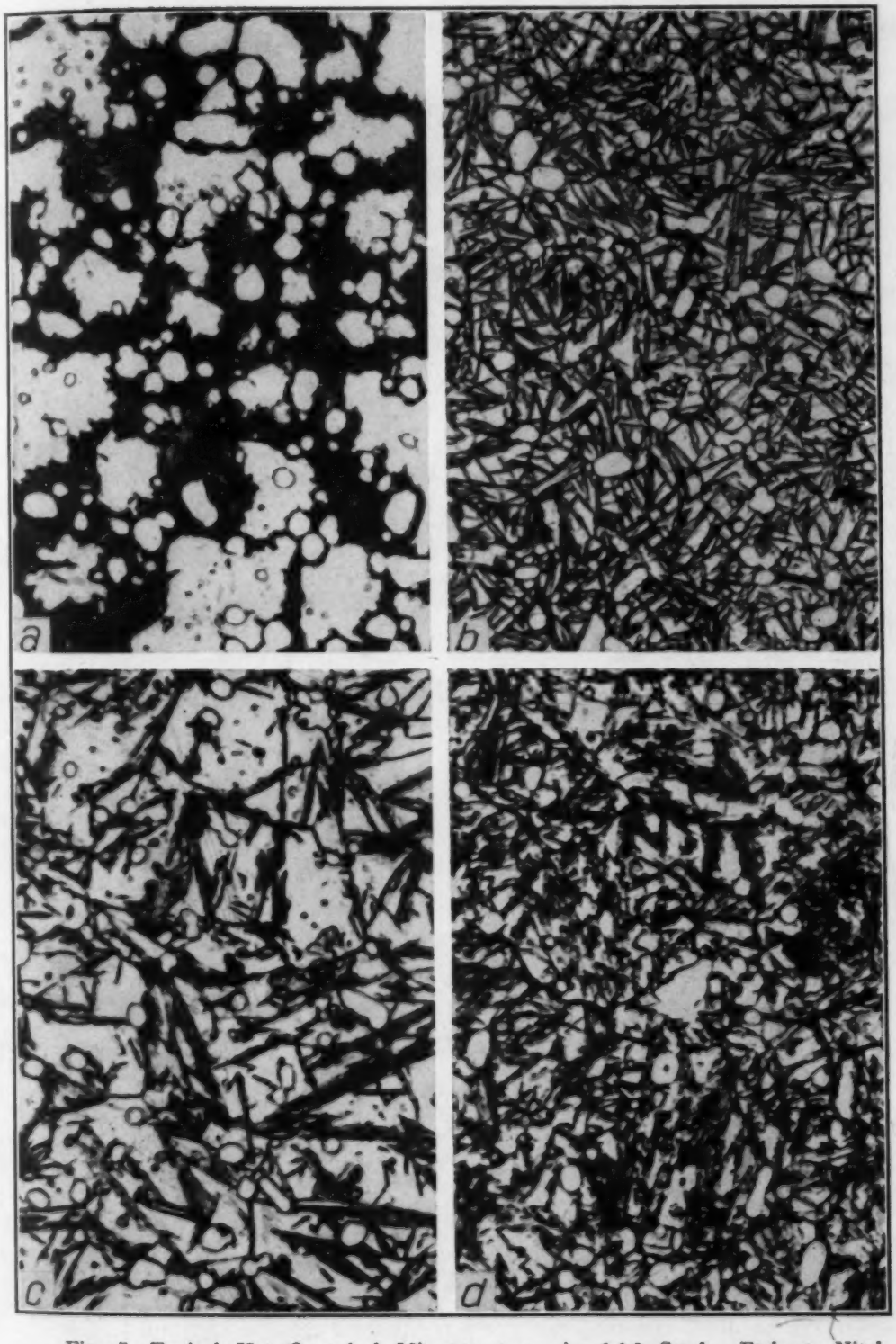


Fig. 7—Typical Hot Quenched Microstructures in 6-6-2 Steel. Etchant—Nital. × 1000. 7a—Hot Quenched to 1230 Degrees Fahr. for 24 Hours. 7b—Hot Quenched to 600 Degrees Fahr. for 24 Hours. Tempered 5 Seconds at 1050 Degrees Fahr. Before Cooling to Room Temperature. 7c—Hot Quenched to 260 Degrees Fahr. for 10 Seconds. Tempered 5 Seconds at 1050 Degrees Fahr. Before Cooling to Room Temperature. 7d—Hot Quenched to 212 Degrees Fahr. for 10 Seconds. Tempered 5 Seconds at 1050 Degrees Fahr. Before Cooling to Room Temperature.

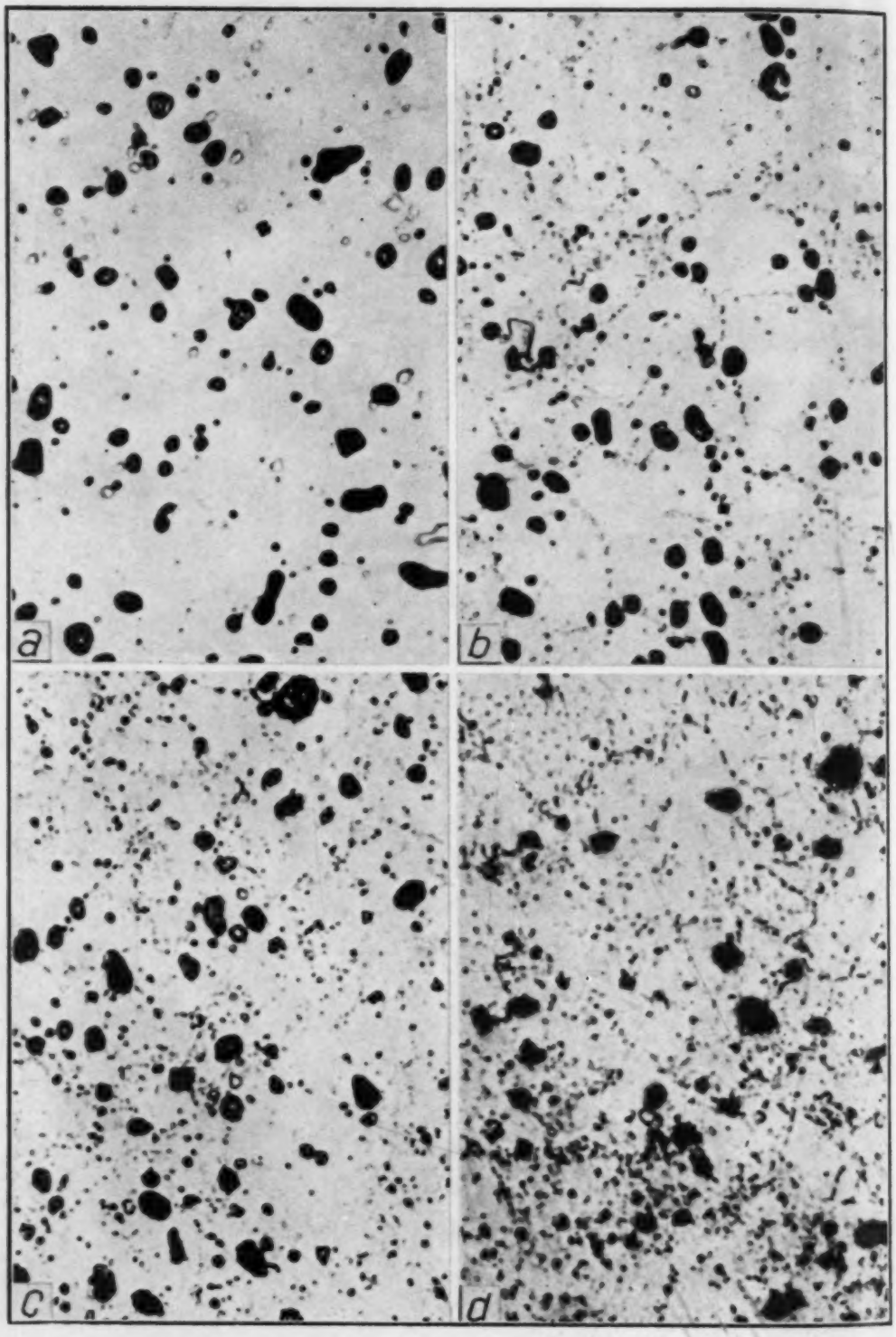


Fig. 8—Carbide Precipitation in 6-6-2 Steel Obtained by Hot Quenching at 1300 Degrees Fahr. Etchant—Murakami's Reagent. × 1000. 8a—10 Seconds at 1300 Degrees Fahr. 8b—5 Hours at 1300 Degrees Fahr. 8c—9 Hours at 1300 Degrees Fahr. 8d—24 Hours at 1300 Degrees Fahr.

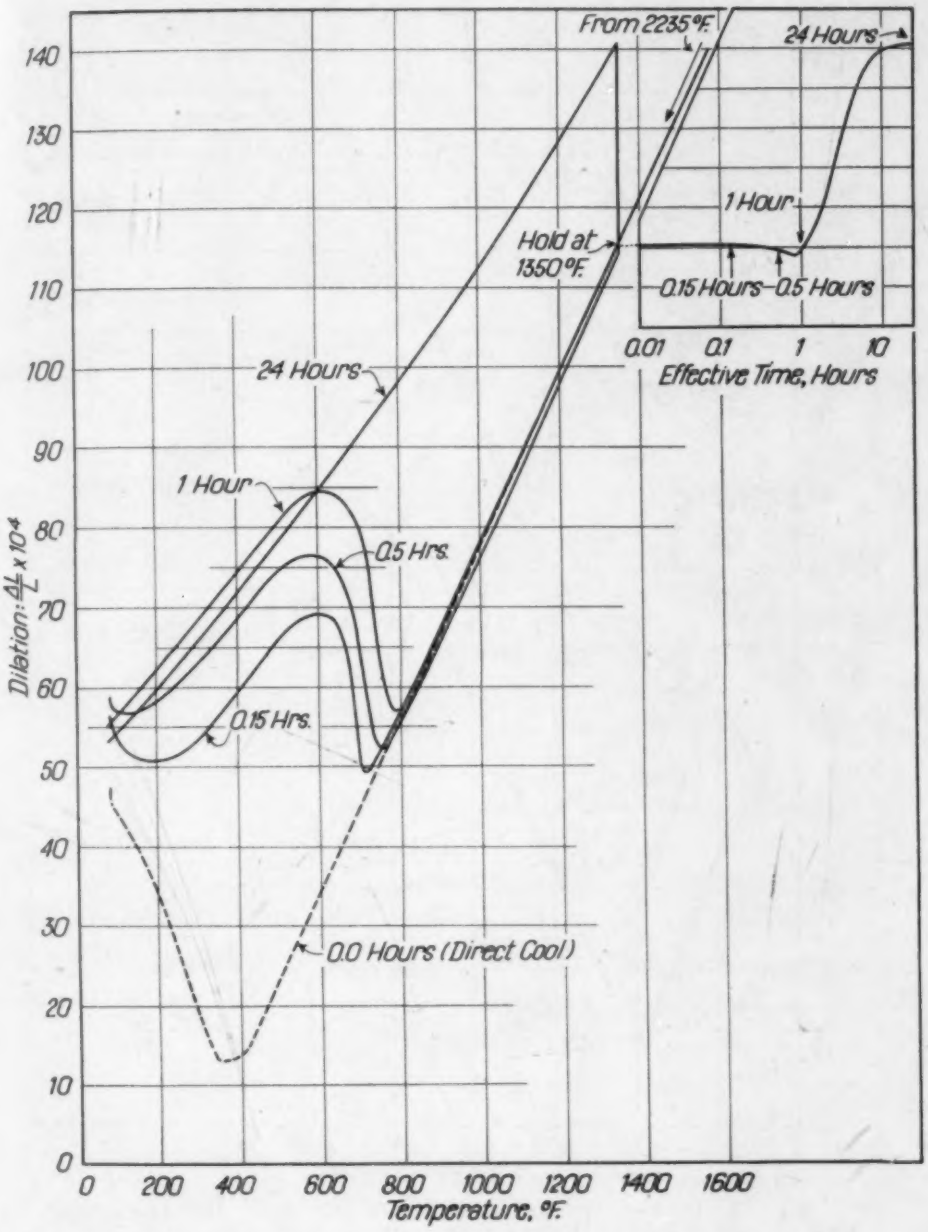


Fig. 9—Dilatometer Curves for 6-6-2. Effect of Hot Quenching Time at 1350 Degrees Fahr.

3. The product of isothermal transformation in the upper range is very fine pearlite, preceded or accompanied by carbide precipitation, and in the lower range, the product is acicular bainite.

4. The martensite transformation sets in on cooling from the high heat temperature when the holding temperature is below 420 degrees Fahr. (215 degrees Cent.) for the 6-6-2 steel and below 430 degrees Fahr. (220 degrees Cent.) for the 18-4-1. These tem-

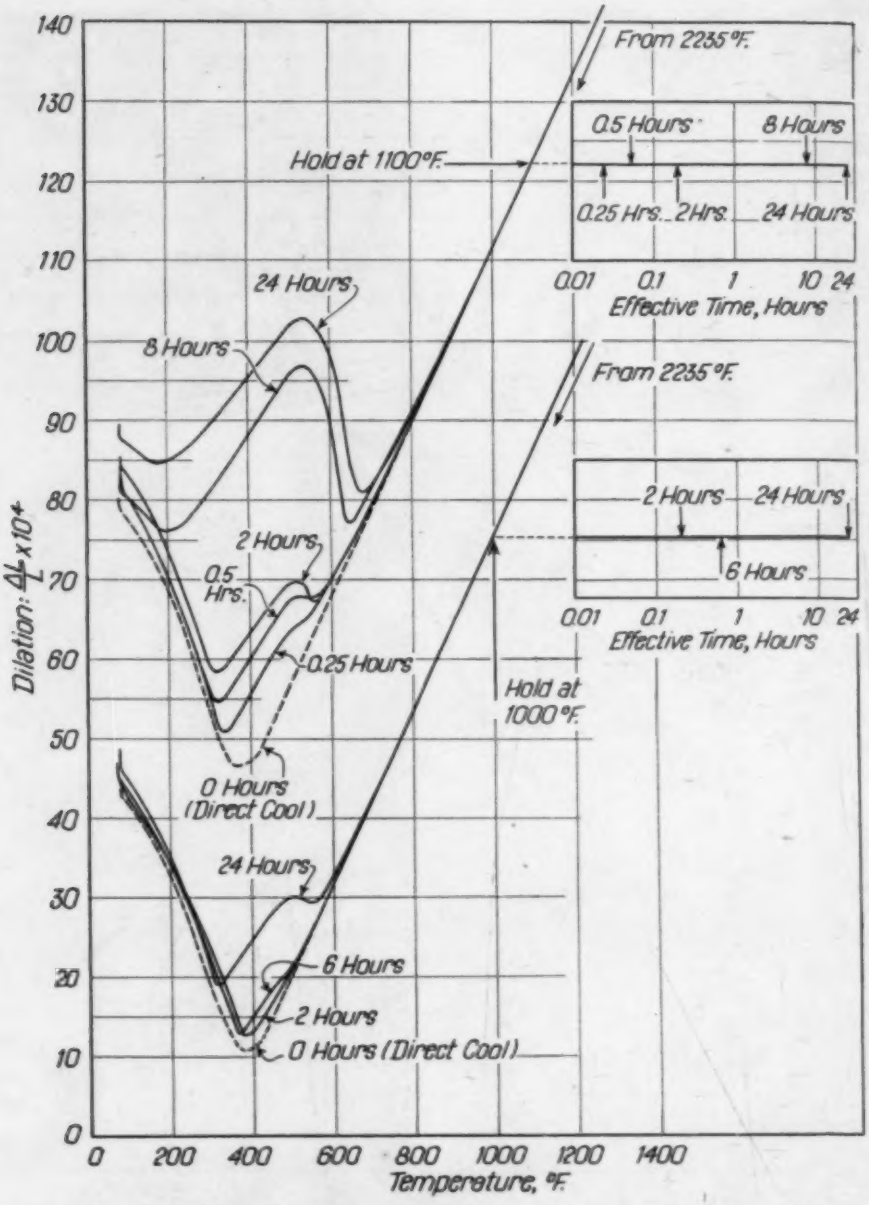


Fig. 10—Dilatometer Curves for 6-6-2. Effect of Hot Quenching Time at 1100 and 1000 Degrees Fahr.

peratures are the M-points of the two steels for the particular austenitizing treatments used.

5. The greater the amount of isothermal transformation in the bainite range above the M-point, the lower is the M-point of the remaining austenite, and the smaller is the amount of martensite formed during subsequent cooling to room temperature. In fact, after 2 hours of isothermal transformation at 600 degrees Fahr. (315 degrees Cent.), the M-point of the remaining austenite has

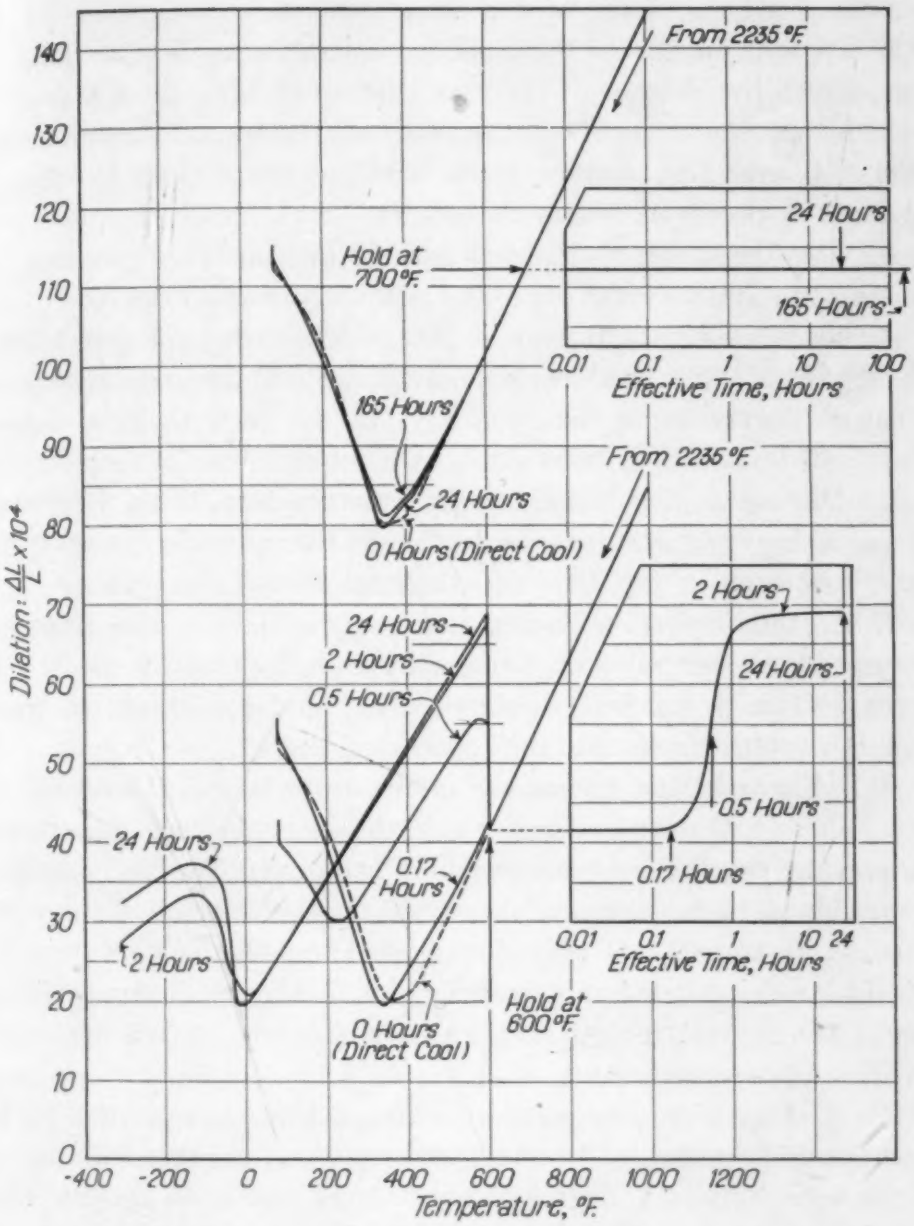


Fig. 11—Dilatometer Curves for 6-6-2. Effect of Hot Quenching Time at 700 and 600 Degrees Fahr.

been lowered to 20 degrees Fahr. (—6.5 degrees Cent.) (cf. Fig. 11) and no martensite forms on cooling to room temperature.

6. Hot quenching to temperatures below the M-point leads to martensite formation between the M-point and the holding temperature followed by isothermal decomposition at the holding temperature. The latter transformation takes place in two stages. The first stage begins immediately on reaching the hot quenching temperature and

proceeds slowly until the second stage sets in with expansion characteristics very similar to those of the bainite formation at temperatures above the M-point. As the hot quenching temperature is lowered, the time interval for the onset of the second stage becomes longer. Below 350 degrees Fahr. (175 degrees Cent.), only the first stage is observed within 24 hours.

7. No detectable isothermal transformation takes place on hot quenching to 700 to 1100 degrees Fahr. (370 to 595 degrees Cent.) for 24 hours. The treatments at 700 to 900 degrees Fahr. (370 to 480 degrees Cent.) produce little influence on the austenite transformation during subsequent cooling, but the 1000 to 1100 degrees Fahr. (540 to to 595 degrees Cent.) treatments exert a very striking effect. During cooling, a split transformation occurs: the higher the hot quenching temperature, the higher is the inception temperature of the first step in the split transformation and the greater is the extent of transformation during this step, while the inception temperature of the second step (originally the M-point of the regular martensite transformation) becomes lower and the extent of transformation occurring in this step decreases.

8. When the hot quenching temperature is raised to 1200 degrees Fahr. (650 degrees Cent.) and above, isothermal transformation into fine pearlite is encountered. During cooling after a 24-hour hot quench at 1200 degrees Fahr. (650 degrees Cent.), the remaining austenite transforms only during the first of the two steps described above. After hot quenching for 24 hours at temperatures above 1200 degrees Fahr. (650 degrees Cent.), no cooling transformation is observed at all.

9. The carbide precipitation which takes place at the higher temperatures is evidenced by the contraction in the isothermal curves (cf. the 1350 and 1400 degrees Fahr. curves in Fig. 4 and the 1400 degrees Fahr. curve in Fig. 6) and by the photomicrographs of Fig. 8. The rate of carbide precipitation increases continuously with the hot quenching temperature whereas the rate of pearlite formation is a maximum at 1320 degrees Fahr. (715 degrees Cent.) for the 6-6-2 steel and at 1350 degrees Fahr. (730 degrees Cent.) for the 18-4-1. In general, however, these two processes overlap.

Effect of Hot Quenching Time—The importance of time as a variable in the hot quenching process is illustrated by the dilatometer curves in Figs. 9, 10 and 11. In each case, the dotted curve shows

the dilation characteristics during a direct quench, i.e., with zero holding time at any given temperature. In Fig. 9, the holding of 6-6-2 steel for only 10 minutes at 1350 degrees Fahr. (730 degrees Cent.) causes no observable isothermal transformation, but during subsequent cooling, the austenite decomposes in two steps as described previously in items 7 and 8. With increasing time of holding at 1350 degrees Fahr. (730 degrees Cent.), the higher step becomes more prominent at the expense of the lower step, but ultimately both steps disappear because the austenite is consumed by the isothermal transformation at 1350 degrees Fahr. (730 degrees Cent.) (see 24-hour curve).

Holding at 1100 and 1000 degrees Fahr. (595 and 540 degrees Cent.) (Fig. 10) where no isothermal transformation occurs even in 24 hours, a period of only 15 minutes at 1100 degrees Fahr. (595 degrees Cent.) or 6 hours at 1000 degrees Fahr. (540 degrees Cent.) is sufficient to cause the split transformation during subsequent cooling. As the holding time at these two temperatures is lengthened, the inception temperature of the first step is raised, and the amount of austenite transforming therein is increased, while the inception temperature of the second step is lowered and the corresponding transformation is decreased (cf. item 7, page 180). It is worth emphasizing once more that no detectable isothermal decomposition takes place during the holding time variations which produce these pronounced effects on subsequent cooling.

At a hot quenching temperature of 700 degrees Fahr. (370 degrees Cent.), where again no isothermal transformation is observed even up to 165 hours, the temperature is apparently too low to have much influence on the subsequent cooling transformations (Fig. 11) although the trend described above is still present. Hot quenching to 600 degrees Fahr. (315 degrees Cent.) introduces new effects because of the isothermal bainite transformation in this range. The longer the holding time at 600 degrees Fahr. (315 degrees Cent.) and the greater the extent of isothermal transformation, the lower is the inception temperature of the martensite during subsequent cooling. This inception temperature corresponds to the M-point of the remaining austenite since there is no split transformation now, the upper step having disappeared. After 2 hours at 600 degrees Fahr. (315 degrees Cent.) the M-point lies below room temperature.

Derivation of Quantitative Transformation Curves—In translat-

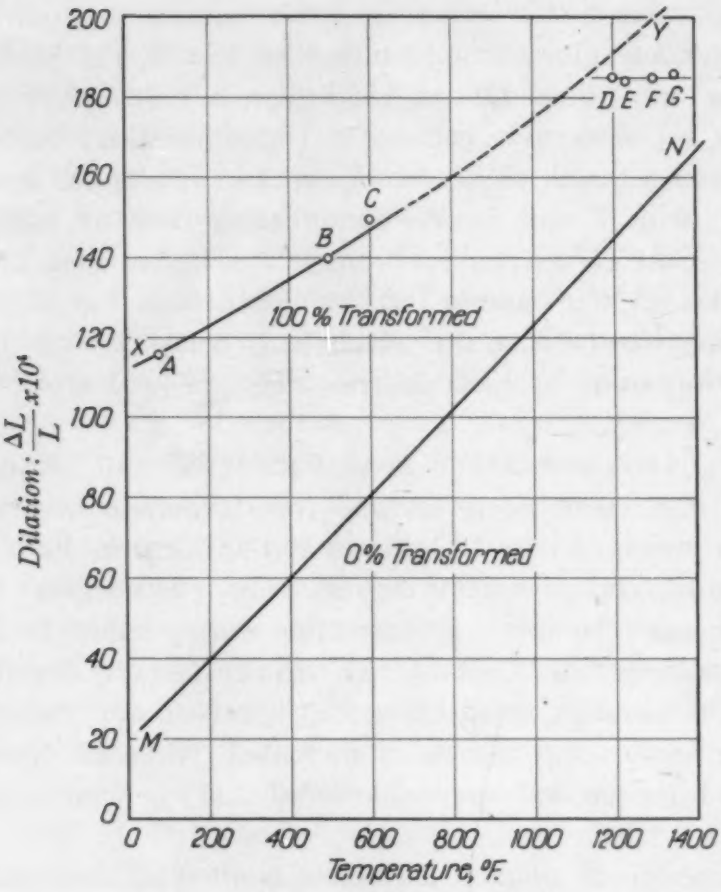


Fig. 12—Calibration Chart for 6-6-2.

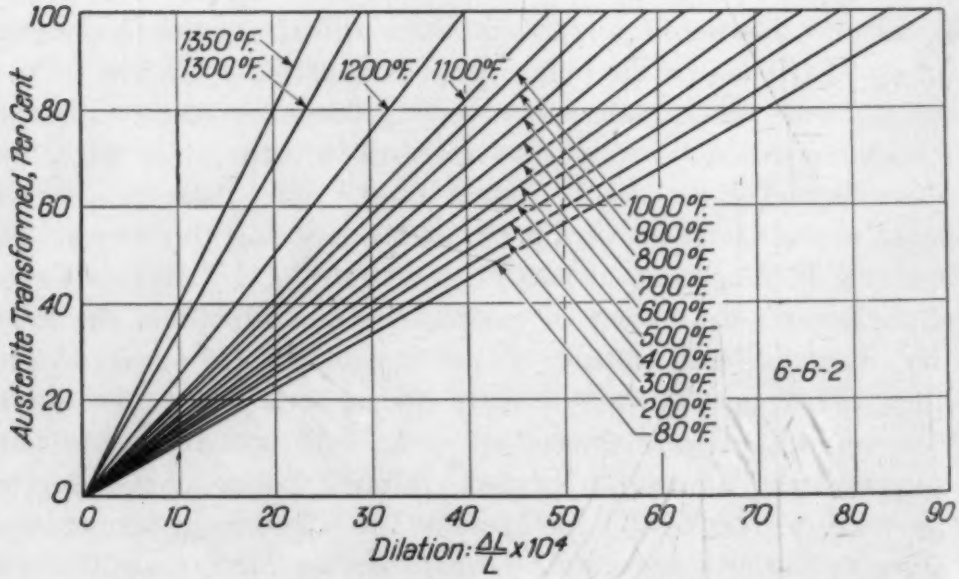


Fig. 13—Per Cent Transformation-Dilation-Temperature Chart for 6-6-2.

ing dilatometric data into transformation curves, it has usually been the practice to take the maximum dilation at a given holding temperature as corresponding to 100 per cent transformation. Then fractional amounts of transformation are calculated on a proportional basis. In high speed steel, however, the isothermal bainite transformations do not go to completion[2] and the change in length measured to where the dilatometer curve "reaches saturation" then corresponds to appreciably less than 100 per cent transformation. Any fractional amount of such a measurement would not give the per cent transformation of all the austenite, but rather the per cent transformation of only the austenite which will transform at the temperature in question.[3] This procedure is somewhat arbitrary, and suffers from the disadvantage that results at various temperatures cannot be compared on a common basis since different amounts of maximum transformation and different expansion characteristics are simultaneously involved.

In the present work, it was considered essential to determine the extent of isothermal transformation on an absolute basis so that quantitative transformation curves could be derived. The method of this procedure is shown in Fig. 12. MN is the cooling-contraction curve of 6-6-2 steel in the austenitic state (with undissolved carbides) during cooling from the high heat temperature of 2235 degrees Fahr. (1225 degrees Cent.). This line represents the average of about 50 runs down to the M-point of 420 degrees Fahr. (215 degrees Cent.) and is extrapolated from there down to room temperature. Similarly, curve XY is the cooling-contraction curve of the completely transformed 6-6-2 steel (i.e., quenched and tempered steel). The two curves have been displaced vertically with respect to each other so that the ordinate between them measures 88.5×10^{-4} inches per inch at room temperature (point A). This is the total change in length, referred to room temperature, exhibited by the austenite in 6-6-2 steel when hardened and tempered to attain complete transformation. For 18-4-1, this expansion was found to be 85.0×10^{-4} inches per inch. Now, if the various transformation products had substantially the same specific volume when referred to room temperature, then the vertical distance between the lines MN and XY in Fig. 12 at any temperature would give the expan-

[2]This phenomenon may be quite general, but is particularly noticeable in the alloy steels.

[3]Once the length becomes constant, no further isothermal transformation is observed even though the run is continued for prolonged periods of time.

sion for 100 per cent transformation at the temperature in question. Observed dilations could then be compared to the proper 100 per cent expansion value to determine fractional amounts of transformation.

It was obviously necessary to check, by independent means, the extent to which this method was justifiable. Consequently, dilatometer specimens that had been partially transformed at 500, 600, 1200, 1230, 1300 and 1350 degrees Fahr. (260, 315, 650, 665, 705 and 730 degrees Cent.) were subjected to the metallographic point-counting technique for ascertaining the per cent of transformation (previously described on page 169). These transformations were then compared to the respective isothermal expansions observed on the specimens, and found to be in excellent agreement with the values predicted by Fig. 12 at 500 and 600 degrees Fahr. (260 and 315 degrees Cent.), but in very poor agreement at 1200 to 1350 degrees Fahr. (650 to 730 degrees Cent.). For example at 500 degrees Fahr. (260 degrees Cent.) the point-counting technique showed 57 per cent bainite transformation on undergoing an isothermal expansion of 40.0 x 10^{-4} inches per inch. Hence, 100 per cent transformation should cause an expansion of 70.0 x 10^{-4} inches per inch. This value when plotted above the curve MN at 500 degrees Fahr. (260 degrees Cent.) falls very close to curve XY at B. Points C, D, E, F and G were similarly calculated and plotted. It is evident from the position of the last four points that the XY curve cannot be safely employed in the range above 1100 degrees Fahr. (595 degrees Cent.) because the transformation products are much denser than those formed at the lower temperatures. This is largely due to the precipitation of heavy alloy carbides which accompanies the pearlite formation. However, the position of the D-G line above the MN line now gives the actual expansions associated with complete transformation in this range, and may be used just like the line ABC for the lower range. No transformation is found in the intermediate range of 700 to 1100 degrees Fahr. (370 to 595 degrees Cent.), and hence no concern need be felt for the validity of the XY line in this region. The good correlation in the lower temperature range suggests that as a first approximation, bainite and martensite in high speed steel have about the same specific volume when compared at the same temperature. (Accurate specific volume measurements have shown that the bainite is actually denser than

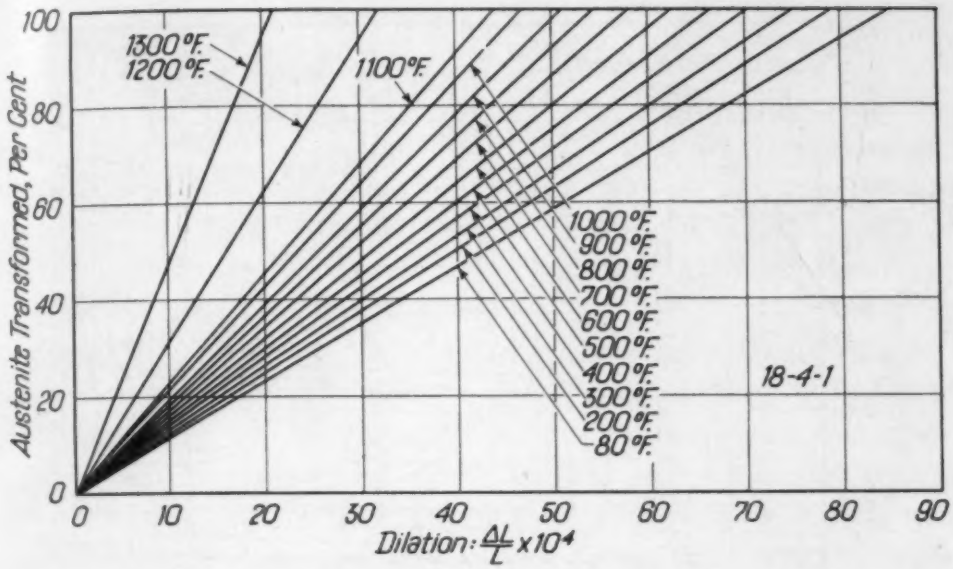


Fig. 14—Per Cent Transformation-Dilation-Temperature Chart for 18-4-1.

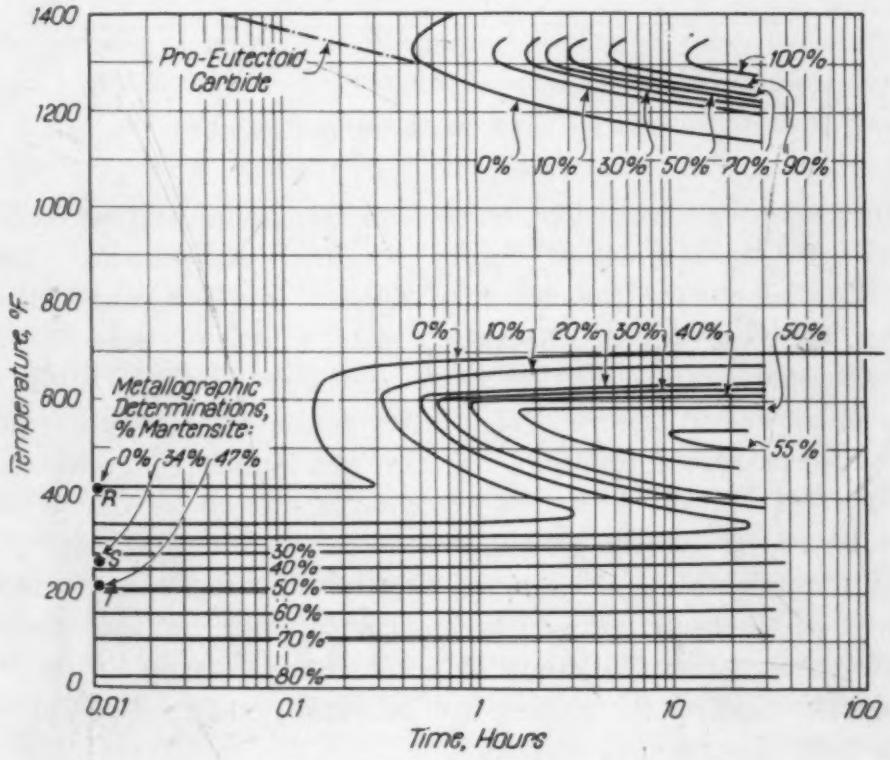


Fig. 15—6-6-2 Transformation Curves.

the martensite (14), but the difference is too small to be considered here.) Consequently, it is also possible to use Fig. 12 in convert-

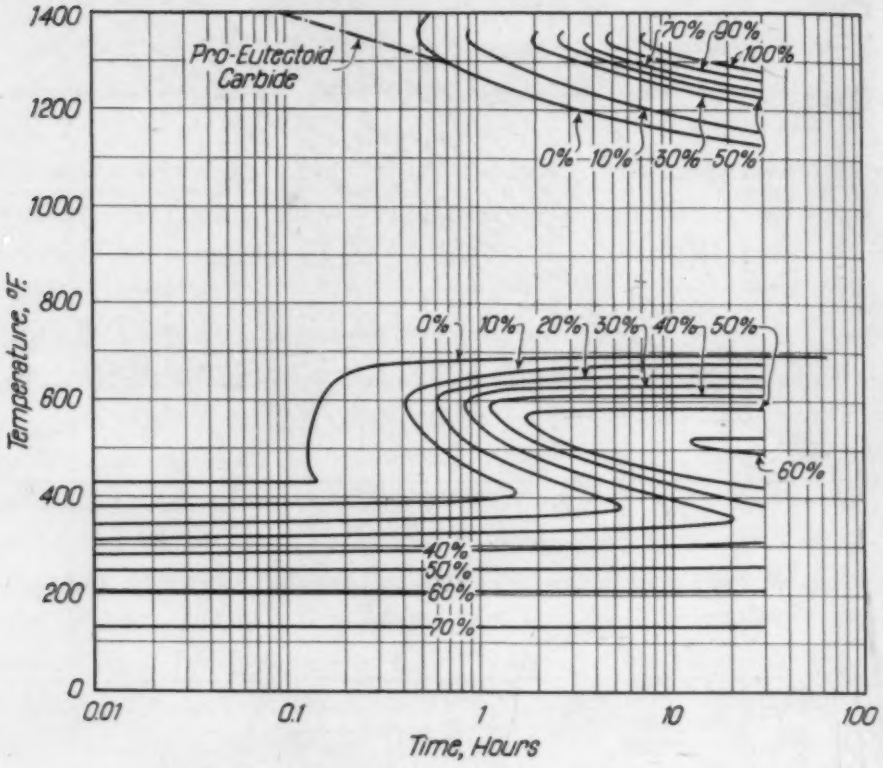


Fig. 16—18-4-1 Transformation Curves.

ing the expansions caused by martensite formation (during cooling) into per cent transformation as a function of the temperature reached.

To facilitate the conversion of dilation into per cent transformation, the data of Fig. 12 were compiled into a per cent transformation-temperature-dilation chart as shown in Fig. 13. In this way, an expansion observed at any temperature is easily converted into per cent transformation. Fig. 14 was similarly developed for 18-4-1 steel. In all cases, the percentages are on a volume basis, and refer to the steel matrix since the excess carbides present remain virtually unaffected. For example, 50 per cent transformation means one-half of the austenitic matrix has decomposed. At zero per cent transformation, there is about 95 per cent of austenite in the steel so that per cent of the matrix can be readily changed to per cent of the total structure, if desired.

With the aid of the calibrations provided by Figs. 13 and 14, the transformation curves for 6-6-2 and 18-4-1 high speed steels were constructed from the appropriate dilatometer curves of Figs. 3 to 6. The transformation curves are shown in Figs. 15 and 16.

Discussion of Results

Transformation Curves—While the transformation curves in Figs. 15 and 16 are commonly called S-curves, they are more accurately described as consisting of two families of C-curves, with the upper part of the first family preceded by proeutectoid carbide precipitation and with the lower part of the second family intercepted by a set of almost horizontal lines.

The transformation represented by the upper C-curves goes to completion in a regular manner if the holding time is sufficiently prolonged. The product is very fine pearlite, although this term is applied in a loose sense in view of the fact that a lamellar structure was not found. At 1400 degrees Fahr. (760 degrees Cent.), where resolution is possible, the ferrite-carbide aggregate appears spheroidal rather than plate-like. The proeutectoid carbide precipitation occurs preferentially along the austenite grain boundaries. (cf. Fig. 8.)

The bainite transformation corresponding to the lower C-curves does not go to completion; it stops even though there is much austenite available for further transformation. For example, in the 6-6-2 steel, 55 per cent of bainite was the most that could be formed isothermally, and in the 18-4-1, the maximum amount was 60 per cent. Yet the bainite transformation actually starts considerably sooner than does the pearlite transformation. Above the nose of the lower C-curves, the rate of bainite formation decreases quite abruptly. At 650 degrees Fahr. (345 degrees Cent.), bainite starts to form in both steels in about 0.2 hour, while at 700 degrees Fahr. (370 degrees Cent.) no bainite appears within 165 hours.

The third range of austenite activity occurs below 420 to 430 degrees Fahr. (215 to 220 degrees Cent.) due to the formation of martensite during cooling, and overlaps the isothermal bainite transformation. The left-hand intercepts of the horizontal lines in this third range are drawn to show the per cents of martensite formed as a function of temperature during cooling below the M-point. These amounts are based on the dilatometric observations, and points R, S and T in Fig. 15 determined metallographically on small rapidly hot quenched specimens demonstrate the justification of this procedure. On cooling directly to room temperature, approximately 75 per cent of the austenite decomposes into martensite, and about 15 per cent more decomposes on immediate cooling to —310 degrees

Fahr. (—190 degrees Cent.). There is no evidence to indicate an incubation period for the martensite formation as was found for the pearlite and bainite formations. However, in the martensite range, the transformation lines are not quite horizontal since isothermal transformation takes place on holding in this range (cf. Figs. 3 and 5). The first stage of this isothermal transformation sets in as soon as the cooling stops, and proceeds very slowly. It is relatively unimportant compared to the preceding cooling transformation and the second isothermal stage, and results in a slight upward slope of the transformation lines in this low temperature range. When the second stage sets in, these lines meet the corresponding bainite lines coming down from the higher temperatures.

In order to establish the nature of the isothermal products which form on holding within the martensite range, the Greninger-Troiano metallograhphic technique was applied to small specimens hot quenched for different lengths of time at different temperatures below the M-point. Fig. 17 illustrates some typical results obtained at 330 degrees Fahr. (165 degrees Cent.). About 10 per cent martensite is found in the structure on reaching this temperature, but no further increase in the amount of this product is observed on isothermal holding. Instead, occasional needles of bainite appear in the austenite surrounding the martensite. Both the martensite and bainite are acicular, but they are readily distinguished from each other, as shown in Fig. 17. The bainite thus formed is small in amount, and nicely accounts for the first stage of isothermal transformation indicated by the dilatometer. With continued holding at 330 degrees Fahr. (165 degrees Cent.), however, the bainite suddenly begins to appear in abundance, and corresponds to the second stage of isothermal transformation. It is clear, therefore, that this second stage is identical, both kinetically and structurally, with the regular bainite transformation that occurs above the M-point. On the other hand, the first stage does not appear above the M-point, and may be regarded as premature bainite formation due to the presence of the martensite formed during the cooling. This bainite may be nucleated by the presence of the martensite, or by the martensite transformation stresses. If it were not for this incidental formation of bainite, the first isothermal stage would be absent, and the martensite transformation lines would be quite horizontal, as was predicted for plain carbon steel two years ago (15). Such a modi-

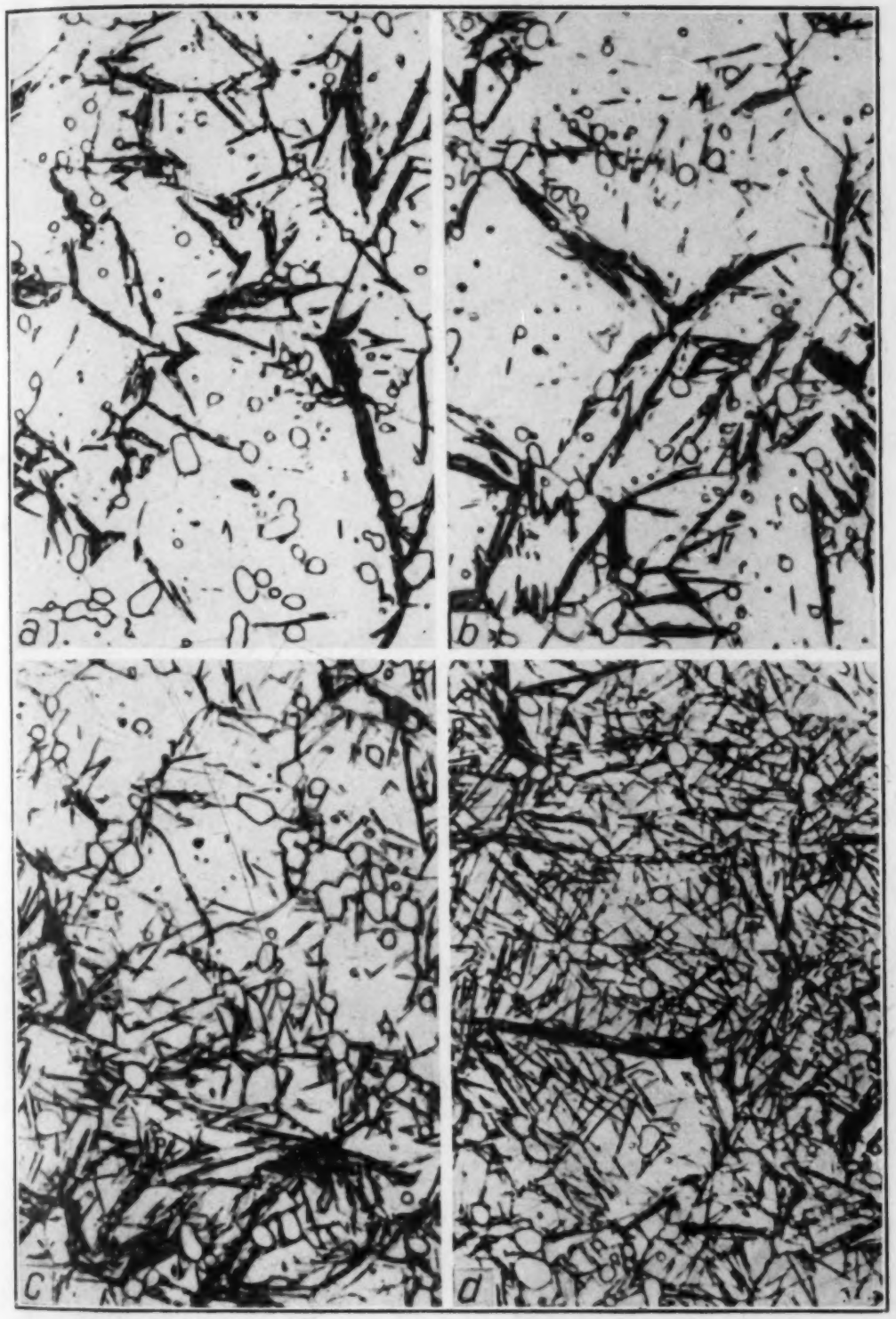


Fig. 17—Microstructures Obtained by Hot Quenching 6-6-2 Steel to 330 Degrees Fahr. and then Tempering 5 Seconds at 1050 Degrees Fahr. Before Cooling to Room Temperature. Etchant—Nital. × 1000. 17a—10 Seconds at 330 Degrees Fahr. 17b—2 Hours at 330 Degrees Fahr. 17c—5 Hours at 330 Degrees Fahr. 17d—24 Hours at 330 Degrees Fahr.

fied S-curve is also in accord with the work of Greninger and Troiano (13) and of Carpenter and Robertson (16) on the kinetics of the austenite-martensite transformation.

The isothermal transformation which occurs at room temperature remains somewhat obscure in origin. Fast rates of cooling to room temperature seem to inhibit subsequent austenite decomposition at room temperature, but in any case, the amount is never greater than 2 to 3 per cent. Such room temperature changes may occur after direct cooling from the high heat treatment or after hot quenching, but only if martensite transformation is under way as room temperature is reached. In this respect and kinetically speaking, the room temperature transformation is similar to the first stage of the isothermal transformation observed on hot quenching below the M-point, and hence the product is believed to be bainite. However, the amount is too small and the co-existing martensite is too copious to allow a metallographic substantiation on this point.

The high degree of stability of the austenite between 700 and 1100 degrees Fahr. (370 and 595 degrees Cent.) is very impressive, but is probably no different in kind from that observed for the lower alloy steels whose transformation curves contain two noses separated by a bay of more or less depth (2). However, in the present case, the bay is sufficiently pronounced to differentiate positively between the pearlite and bainite transformations. It seems quite reasonable to assign the two sets of C-curves to different types of nucleated reactions as has been done with the simpler steels (17) and cast irons (18), the upper reaction being nucleated by carbide and the lower reaction by ferrite.

Cooling Transformations After Hot Quenching—As the dilatometer curves have demonstrated, the austenite which does not decompose during the hot quenching operation becomes available for transformation on cooling to room temperature. The details of these cooling transformations have been described in a previous section. Figs. 18 and 19 show the temperature ranges covered by these transformations on cooling at 5 to 10 degrees Fahr. per minute after hot quenching for 24 hours. The split transformation range after 24 hours at 700 to 1100 degrees Fahr. (370 to 595 degrees Cent.) is evident. In the light of the transformation curves in Figs. 15 and 16, it now appears that the higher transformation product is bainite and the lower product is martensite. Only bainite forms after cool-

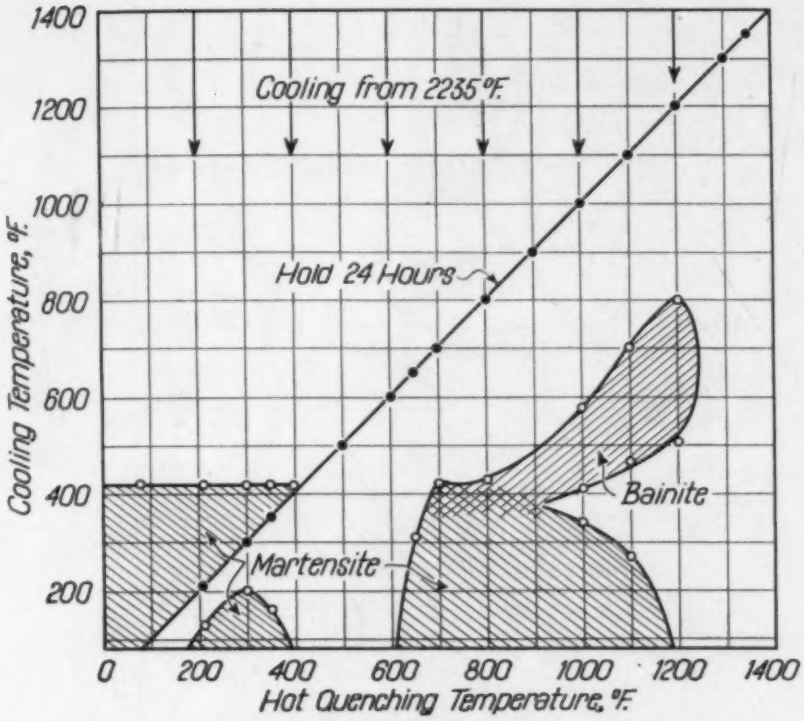


Fig. 18—Transformation Ranges in 6-6-2 During Cooling Before and After Hot Quenching. Holding Time—24 Hours. Rate of Cooling After Hot Quenching—5 to 10 Degrees Fahr. Per Minute.

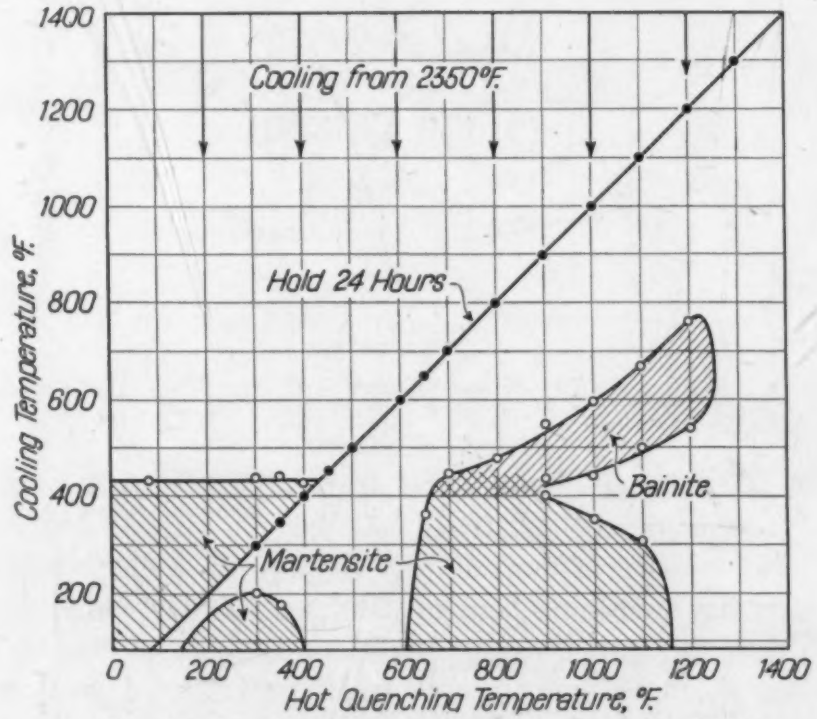


Fig. 19—Transformation Ranges in 18-4-1 During Cooling Before and After Hot Quenching. Holding Time—24 Hours. Rate of Cooling After Hot Quenching—5 to 10 Degrees Fahr. Per Minute.

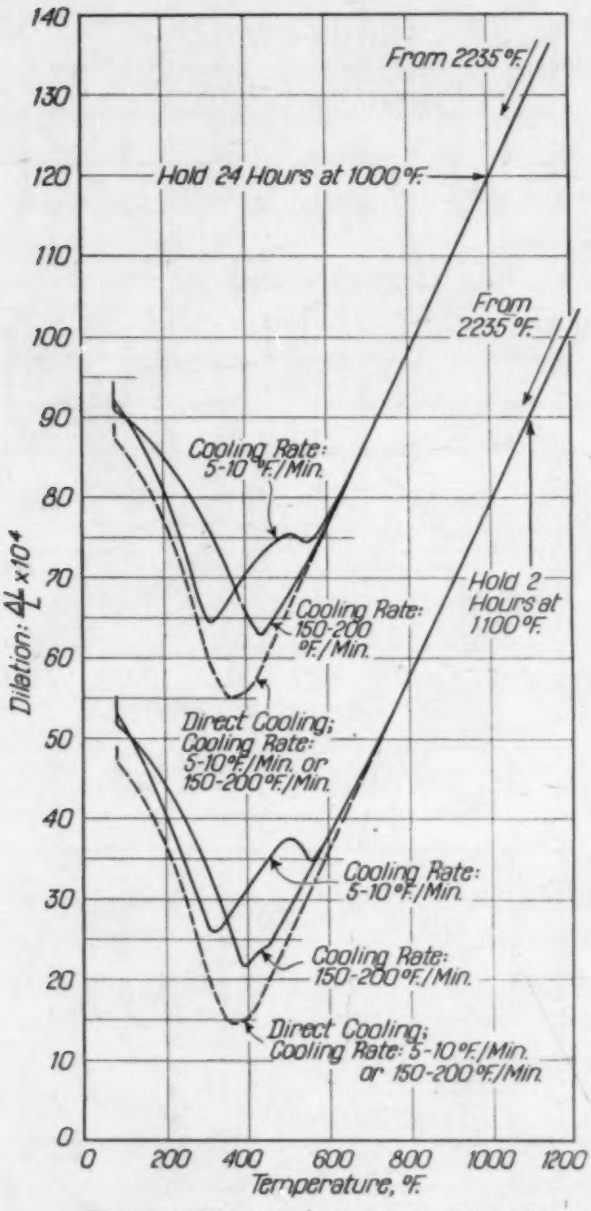


Fig. 20—Effect of Cooling Rate After Hot Quenching to 1000 and 1100 Degrees Fahr. for 24 Hours.

ing from 1200 degrees Fahr. (650 degrees Cent.) while only martensite forms after cooling from 650 degrees Fahr. (345 degrees Cent.). No transformation during cooling to room temperature occurs after hot quenching between 600 and 400 degrees Fahr. (315 and 205 degrees Cent.) because sufficient isothermal decomposition takes place at these temperatures to depress the M-point of the remaining austenite below room temperature.

As might be suspected, bainite transformation during cooling

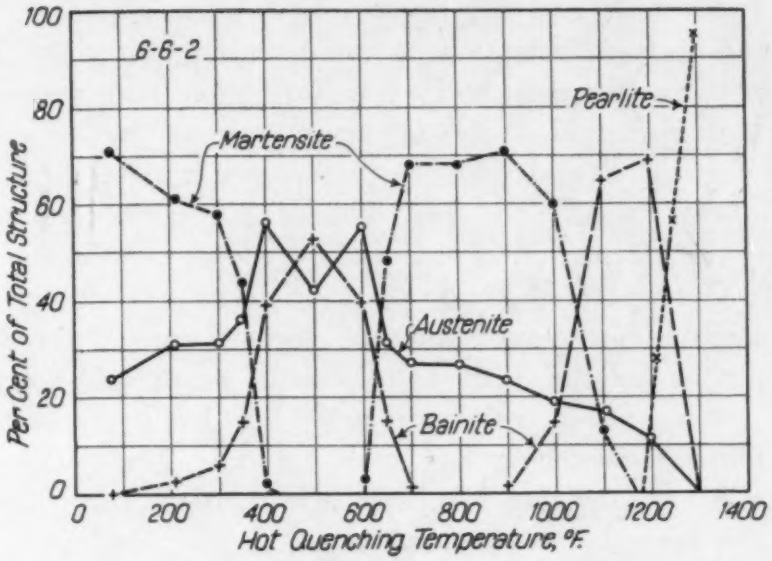


Fig. 21—Structural Composition of 6-6-2 at Room Temperature as a Function of Hot Quenching Temperature. Time of Holding—24 Hours. Rate of Cooling from Holding Temperature —5 to 10 Degrees Fahr. Per Minute.

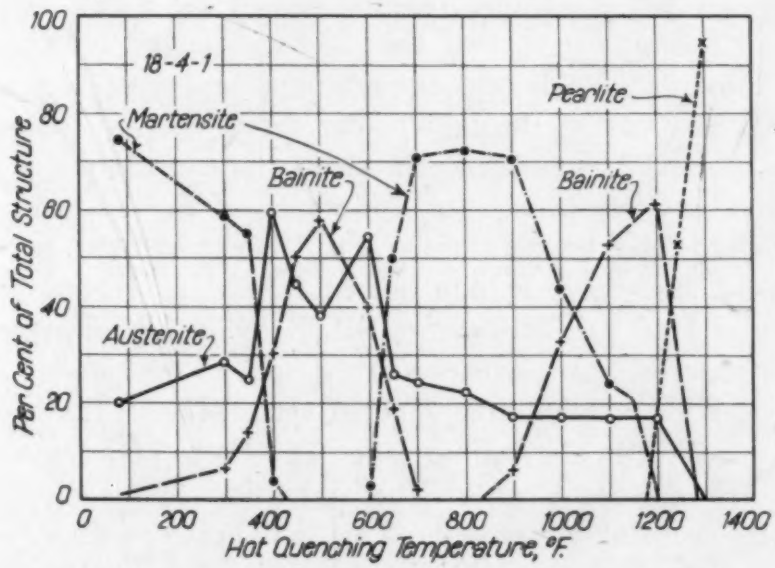


Fig. 22—Structural Composition of 18-4-1 at Room Temperature as a Function of Hot Quenching Temperature. Time of Holding—24 Hours. Rate of Cooling from Holding Temperature —5 to 10 Degrees Fahr. Per Minute.

after hot quenching is greatly affected by the rate of cooling. In fact, it can be partially or even completely suppressed by rapid cooling. This is demonstrated by Fig. 20 in which two rates of cooling (5 to 10 degrees Fahr. per minute and 150 to 200 degrees Fahr. per minute) are compared after hot quenching to 1000 and 1100

degrees Fahr. (540 and 595 degrees Cent.) for 24 hours. Inhibiting the bainite transformation by rapid cooling raises the inception temperature of the martensite transformation and permits more martensite to form. It is also important to note that on cooling at the slow rate of 5 to 10 degrees Fahr. per minute directly from the high heat temperature, i.e., without hot quenching, only the martensite transformation is observed. In other words, hot quenching at 1000 to 1100 degrees Fahr. (540 to 595 degrees Cent.) (even though it causes no isothermal decomposition) has the effect of shifting the bainite C-curves to shorter times, and much faster rates of cooling are necessary to avoid the bainite transformation than if the hot quenching had been omitted. The details of this phenomenon are now under further investigation, but tentatively it is proposed here that high speed steel hot quenched into the bay of the S-curve undergoes a nucleation process or an atomic rearrangement within the austenite such as probably occurs at the higher or lower temperatures where the austenite is found to transform isothermally. Such pretransformation changes at a bay temperature might well accomplish part of the incubation stage for the formation of bainite at the lower temperatures, and thus accelerate the bainite transformation during cooling from the bay temperature.

Figs. 21 and 22 summarize the structural constitution of the two steels after hot quenching for 24 hours at various temperatures and cooling to room temperature at 5 to 10 degrees Fahr. per minute. In the 6-6-2 steel more bainite can be formed on cooling after hot quenching at 1100 to 1200 degrees Fahr. (595 to 650 degrees Cent.) than by isothermal transformation at the nose of the bainite C-curves. In both steels the maximum amount of retained austenite is obtained by hot quenching in the bainite range of 400 to 600 degrees Fahr. (205 to 315 degrees Cent.). The formation of bainite seems to stabilize the remaining austenite. This is indicated both by the self-stopping of the isothermal bainite formation long before the austenite is completely consumed and by the pronounced lowering of the M-point of the remaining austenite. Both of these effects may be combined to yield as much as 60 per cent of retained austenite in the steel at room temperature.

Practical Considerations

The common practice of hot quenching high speed steel into

the range of 1000 to 1400 degrees Fahr. (540 to 760 degrees Cent.) as a part of the hardening operation should now be viewed in the light of the transformation curves of Figs. 15 and 16. The most rapid rate of pearlite formation occurs at 1320 degrees Fahr. (715 degrees Cent.) in the 6-6-2 steel and at 1350 degrees Fahr. (730 degrees Cent.) in the 18-4-1. In each case this transformation begins in approximately 30 minutes. However, above 1300 degrees Fahr. (705 degrees Cent.) carbide precipitation precedes the pearlitic transformation and at 1400 degrees Fahr. (760 degrees Cent.) this precipitation is first observable in 3 minutes with 6-6-2 and in 5 minutes with 18-4-1. The possible occurrence of these transformations during hot quenching, particularly in the case of large tools, has not been fully appreciated, and may lead to inferior properties.

Hot quenching in the range of 1000 to 1100 degrees Fahr. (540 to 595 degrees Cent.), where isothermal decomposition does not take place, may also introduce hitherto unsuspected effects because the steel is thereby conditioned for bainite formation at the expense of martensite during the cooling to room temperature. The amount of bainite thus formed increases as the cooling rate decreases, so that air cooling of large tools from the hot quench may result in appreciable quantities of bainite in the final structure. Further work is necessary to establish whether this bainite is beneficial or deleterious in tool and die applications, but once this is done, the present work provides the necessary principles of austenite decomposition for obtaining more or less bainite, as desired.

Since high speed steel can be heat treated to yield bainite and retained austenite in controlled amounts far in excess of the percentages encountered in regular practice, it would be desirable to ascertain whether the practical utility of high speed steel can be appreciably extended by heat treating to develop judicious mixtures of martensite, bainite, and retained austenite. However, such an investigation must await further studies on the tempering characteristics of the hot quenched structures.

Conclusions

1. The kinetics of austenite decomposition in 6-6-2 and 18-4-1 high speed steels have been investigated and found to be virtually alike in every respect.

2. Both steels exhibit two temperature ranges of isothermal activity after definite incubation periods. The product of isothermal transformation in the upper range (1400 to 1200 degrees Fahr.) is very fine pearlite, preceded or accompanied by carbide precipitation. In the lower range (675 to 350 degrees Fahr.), the product is acicular bainite. The pearlite transformation goes to completion if the holding time is sufficiently extended, but the bainite transformation stops long before the austenite is completely exhausted.

3. For the particular austenitizing treatments used, the martensite transformation sets in at 420 degrees Fahr. (215 degrees Cent.) for the 6-6-2 steel and at 430 degrees Fahr. (220 degrees Cent.) for the 18-4-1. There is no observable incubation period prior to the martensite formation once the M-point is reached.

4. Hot quenching to temperatures within the martensite range leads to martensite formation during the cooling from the M-point to the holding temperature, followed by isothermal bainite formation at the holding temperature. The latter transformation takes place in two stages; the first stage consisting of the formation of occasional plates of bainite in the martensitic microstructural regions, and the second stage occurring later with the general formation of bainite throughout the structure.

5. There is no detectable transformation on hot quenching into the intermediate range of 700 to 1100 degrees Fahr. (371 to 595 degrees Cent.). Nevertheless, such hot quenching, particularly above 900 degrees Fahr. (480 degrees Cent.), conditions the austenite for bainite formation at the expense of martensite during subsequent cooling. The cooling transformation takes place in two steps; the product of the upper step being bainite and that of the lower step being martensite. As the holding time and temperature are increased in this hot quenching range, the amount of bainite formed during cooling increases, and leaves less austenite available for martensite formation. The amount of bainite thus formed also depends upon the rate of cooling; rapid cooling tends to suppress the bainite transformation, and hence reserves more austenite for decomposition into martensite. This type of split transformation is likewise evident on cooling after hot quenching in the pearlite region if the holding time is brief enough to avoid the formation of much pearlite.

6. By suitable hot quenching treatments, many combinations of pearlite, bainite, martensite and retained austenite can be secured in 6-6-2 and 18-4-1 steels at room temperature.

7. Attention is called to the relation between the present work and the common practice of hot quenching high speed steel in the range of 1000 to 1400 degrees Fahr. (540 to 760 degrees Cent.) as a part of the hardening operation.

ACKNOWLEDGMENT

The authors are indebted to J. P. Gill and the Vanadium-Alloys Steel Company for permission to publish this work.

Bibliography

1. E. S. Davenport and E. C. Bain, "Transformation of Austenite at Constant Subcritical Temperatures." *Transactions,* American Institute of Mining and Metallurgical Engineers, Iron and Steel Division, 1930, p. 117-154. 154.
2. E. S. Davenport, "Isothermal Transformations in Steel," TRANSACTIONS, American Society for Metals, Vol. 27, 1939, p. 837-886.
3. N. P. Allen, L. B. Pfeil and W. T. Griffiths, "The Determination of the Transformation Characteristics of Alloy Steels," British Iron and Steel Institute, Special Report No. 24, 1939, p. 369-390.
4. I. Bornatsky, "Improving the Resistance of High Speed Tools," *Stal,* 1939, No. 6, p. 44-46.
5. R. H. McCarthy, "Hot-Quenching of High Speed Steel," *Mechanical Engineering,* March, 1942, p. 201-204.
6. B. H. DeLong and F. R. Palmer, "What Happens When High Speed Steel is Quenched," TRANSACTIONS, American Society for Steel Treating, Vol. 13, 1928, p. 420-434.
7. J. L. Ham, R. M. Parke and A. J. Herzig, "Kinetics and Reaction Products of the Isothermal Transformation of a 6 Per Cent Tungsten—6 Per Cent Molybdenum High Speed Steel," TRANSACTIONS, American Society for Metals, Vol. 29, 1941, p. 623-637.

7A. P. Payson, "How Alloying Elements Affect High Alloy Steels," *The Iron Age,* April 18, 1940, p. 31-35.

8. A. Gulyaev, "Decomposition of Austenite in High Speed Steel at Constant Temperature," *Metallurg,* Vol. 15, No. 9, 1940, p. 43-50. "Molybdenum in Low Alloy High Speed Steels," *Stal,* Vol. 10, No. 9, 1940, p. 33-38.
9. S. Steinberg and V. Zyuzin, "Aging of Supercooled Austenite in High Speed Steel," *Metallurg,* Vol. 14, No. 3, 1939, p. 56-64.
10. J. W. Meier, "Dilatometric Measurements of Austenite Transformation in High Speed Steel," Bachelor of Science Thesis, Massachusetts Institute of Technology, May, 1941.
11. D. B. Oakley and J. F. Oesterle, "Dilatometric Studies in the Transformation of Austenite in a Molybdenum Cast Iron," TRANSACTIONS, American Society for Metals, Vol. 28, 1940, p. 832-852.
12. "Tentative Method of Test for Linear Expansion of Metals," *Proceedings,* American Society for Testing Materials, Vol. 34, 1934, p. 733-742.
13. A. B. Greninger and A. R. Troiano, "Kinetics of the Austenite to Martensite Transformation in Steel," TRANSACTIONS, American Society for Metals, Vol. 28, 1940, p. 537-574.
14. R. E. Peterson, "Effects of Isothermal Transformation of Residual Austenite on the Properties of High Speed Steel," Master of Science Thesis, Massachusetts Institute of Technology, May, 1942.

15. Discussion by M. Cohen on paper by A. B. Greninger and A. R. Troiano entitled "Kinetics of the Austenite to Martensite Transformation in Steel," TRANSACTIONS, American Society for Metals, Vol. 28, 1940, p. 563-568.
16. H. Carpenter and J. M. Robertson, "Metals", Oxford University Press, 1939, Vol. II, p. 880-914.
17. R. F. Mehl, "The Physics of Hardenability," American Society for Metals Symposium on "Hardenability of Alloy Steels," 1939, p. 1-65.
18. R. A. Flinn, M. Cohen and J. Chipman, "The Acicular Structure in Nickel-Molybdenum Cast Irons," TRANSACTIONS, American Society for Metals, Vol. 30, 1942, p. 1255.

DISCUSSION

Written Discussion: By K. J. Trigger, assistant professor of mechanical engineering, University of Illinois, Urbana, Ill.

The authors are to be commended for another valuable contribution to the treatment of high speed steel.

The writer has been investigating the effect of holding time at 1025 degrees Fahr. (550 degrees Cent.) on the transformation occurring in 6-6-2 type high speed steel. His results bear out the data presented in this paper.

The method of detecting the austenitic transformation has been described elsewhere,[1] but may be reviewed briefly here. The apparatus consists of a complete magnetic circuit placed within and around an electrically heated furnace. In the magnetic circuit are two coils, one of which acts as an exciter or primary and the other a pickup or secondary. The operation of the apparatus consists of placing the specimen between the magnetic poles (the secondary being movable) and adjusting the current through the primary coil until a fixed secondary voltage of 7 volts is obtained. With a fixed secondary voltage a constant flux density in the magnetic circuit is assured, and variable losses due to steel furnace shell are eliminated.

The principle of operation is based on the change in magnetic permeability that accompanies the change from austenite to its decomposition products. For example, a $\frac{3}{16}$-inch thick sample of high speed steel in the austenitic condition requires a primary current of approximately 1 ampere to give the 7 volts secondary e.m.f. whereas the sample after transformation requires but 0.4 $\pm$ amperes in the primary coil to give 7 volts at the secondary. The furnace containing the magnetic circuit is readily controlled to within $\pm$ 5 degrees Fahr. of the desired holding temperature.

The magnetic method of study admittedly does not consider possible effects due to carbide precipitation, Curie effects or stress relief. However, the magnetic changes brought about by the transformation of austenite are very pronounced and in the opinion of the writer good qualitative indications of the transformation temperatures.

Using the magnetic apparatus the writer has investigated the effect of holding time at 1025 degrees Fahr. (550 degrees Cent.) on the austenitic decomposition of 6-6-2 high speed steel under the following test conditions:

Samples: $\frac{5}{8}$ inch square x $\frac{3}{16}$ inch thick, all decarburization removed.

[1] R. H. McCarthy, "Hot Quenching of High Speed Steel," *Mechanical Engineering*, March 1942, Vol. 64, No. 3, p. 201-204.

Preheat: 10 minutes at 1025 degrees Fahr. in lead covered with charcoal.

High Heat: 1¼ minutes at 2250 ± 5 degrees Fahr. Globar furnace with carbonaceous muffle to minimize scaling.

Hot Quench: 15 seconds in lead at 1025 ± 5 degrees Fahr.

Holding: For desired time in "magnetic" furnace at 1025 ± 5 degrees Fahr.

Cooling to 160 degrees Fahr.: 2 hours ± 3 minutes or an average rate of about 7.5 degrees Fahr. per minute. (Regulated by fan and air jet.)

During the holding time readings of the primary current for 7 volts secondary e.m.f. were taken at regular intervals. During cooling (to 160 degrees Fahr. the lowest temperature) readings of primary current were taken at each 20-degree Fahr. interval. From the data, curves of the change in primary current ($\triangle I_P$) for each corresponding temperature interval ($\triangle T$) were plotted against the mid-temperature of the interval, $\triangle T$. The resulting curves resemble inverse rate cooling curves and clearly show the inception of and maximum rate of the austenite to bainite (or martensite) reaction.

The following effects of holding time at 1025 degrees Fahr. (550 degrees Cent.) were observed:

a) A slight decrease in primary current as the holding time at 1025 degrees Fahr. (550 degrees Cent.) increased from ½ to 24 hours, somewhat more pronounced as time increased. The increased magnetic permeability is attributed to the precipitation of carbides from the austenite.

b) A single transformation (to martensite) beginning at 370 degrees Fahr. (190 degrees Cent.) and peaking at 290 degrees Fahr. (145 degrees Cent.) for continuous cooling from 1025 degrees Fahr. (550 degrees Cent.). No holding time. R_e 64.9. Essentially the same conditions existed after holding 15 minutes at 1025 degrees Fahr. (550 degrees Cent.).

c) A double transformation beginning at 530 degrees Fahr. (275 degrees Cent.) and peaking at 490 degrees Fahr. (slight) followed by a pronounced transformation beginning at 370 and peaking at 310 degrees Fahr. after holding ½ hour at 1025 degrees Fahr. R_e 64.9.

d) A double transformation for all holding times over ½ hour at 1025 degrees Fahr. (550 degrees Cent.), the data tabulated as follows:

Holding Time Hours	Upper (Bainite) Reaction		Lower (Martensite) Reaction		
	Inception	Peak	Inception	Peak	R_e
1	520	500	390	310	64.9
1½	550	500	390	330	65.2
2	570	510	390	330	65.2
4	570	510	390	330	65.9
12	630	530	370	310	66.0
24	640	540	310	290	66.1

As the holding time at 1025 degrees Fahr. (550 degrees Cent.) increased from ½ hour to 24 hours, the intensity of the bainite reaction increased at the expense of the martensite reaction until at 24 hours the martensite reaction had all but disappeared. Compare with item 5, page 178, and also item 7, page 180. Comparing the results of the magnetic method with those of Messrs. Cohen, Gordon, and Rose, under comparable holding temperatures, reveals the following:

Holding Time Hours	Cohen et al. Beginning of Transformation		Magnetic Method			
			Bainite		Martensite	
	Bainite	Martensite	Beginning	Peak	Beginning	Peak
0	...	380±	...	...	370	290
2	500 (?)	380±	570	510	390	330
24	570	310±	640	540	310	290

Notice that the R_c hardness of the quenched samples increases gradually from 64.9 to 66.1 as the holding time at 1025 degrees Fahr. (550 degrees Cent.) increases from 0 to 24 hours. This is believed to be due to precipitation of carbides at 1025 degrees Fahr. (550 degrees Cent.). Multiple tempering, 1025 degrees Fahr. (550 degrees Cent.), 1 hour, air cooled, and 1025 degrees Fahr. (550 degrees Cent.), 1 hour, furnace cooled, erases much of the difference, the variation being from 66.5 to 67.0 with longer holding times on the original quench giving the higher hardnesses.

The industrial significance of the effects of hot quenching time on 6-6-2 high speed steel are yet to be evaluated. The writer hopes to have some test data available before long.

He is grateful for this opportunity to substantiate and to supplement data for one of the hot quenching temperatures.

Written Discussion: By G. M. Butler, research metallurgist, Allegheny Ludlum Steel Corp., Dunkirk, N. Y.

All manufacturers and users of high speed steels should find food for thought in this excellent paper, and the promised future publications covering effect of tempering and mechanical properties will be awaited with interest.

The authors have not stated in so many words that the best temperature range for hot quenching is 700 to 900 degrees Fahr. (370 to 480 degrees Cent.), although their data all point to this conclusion. For example, Figs. 21 and 22 show that hot quenching within this range yields essentially the same ratios of martensite to austenite as does uninterrupted cooling at 5 to 10 degrees Fahr. per minute clear to room temperature, with very little of the presumably undesirable bainite. Do the authors have any reasons to feel that this temperature range is not best for hot quenching?

In commercial hot quenching, soaking times in the hot quench would not be apt to exceed the time necessary for equalization of temperature, which would mean not over 10 or 15 minutes. Fig. 10 shows that even at 1000 to 1100 degrees Fahr. (540 to 595 degrees Cent.) only slight changes occur after 15 minutes in the hot quench. Fig. 11 shows, however, that even shorter times cause considerable change at 600 degrees Fahr. (315 degrees Cent.), which emphasizes the desirability of the commonly used range of 700 to 1100 degrees Fahr. (370 to 595 degrees Cent.) for pieces of considerable size. If all that is desired of the hot quench is reduction of temperature gradients in moderate-sized pieces, a very short holding time at almost any temperature up to at least 1200 degrees Fahr. would seem safe.

Fig. 3 suggests that expansion of high speed steel in tempering could be considerably reduced by hot quenching for a carefully controlled time at perhaps 500 degrees Fahr. (260 degrees Cent.), if the presence of some small amount of tempered bainite were not objectionable. Holding at this temperature causes marked expansion as part of the austenite transforms to bainite, leaving less to cause expansion by transforming to martensite during tempering.

It is probable that the over-all dimensional changes due to hot quenching and tempering would be less than in direct quenching and tempering.

Written Discussion: By M. J. Weldon, metallurgist, Henry G. Thompson & Son Company, New Haven, Conn., and D. G. Watson, staff metallurgist, Sanderson Works, Crucible Steel Company of America, Syracuse, N. Y.

We feel fully confident that this splendid contribution to the technical literature will serve in large measure to direct concerted attention to the so-called "no man's land" that high speed steel tools pass through during the course of heat treatment. We refer to that period beginning at the moment the steel leaves the superheating furnace and terminating upon its placement in the tempering chamber. Ever since the time that Taylor and White[2] advocated the hot quenching of high speed steel tools into molten lead at 1150 degrees Fahr. (620 degrees Cent.) followed by cooling in air to room temperature prior to tempering, multifarious forms of this general principle have been employed with excellent results. We see the idea applied every day in such forms as the "interrupted quench" where tools are removed from the oil quench after the surfaces have lost their heat color and permitted to cool in air to room temperature. As the authors have pointed out, we also witness the applied principle in the use of certain types of salt baths where hot quenching and surface descaling may be carried out simultaneously.

It has seemed to us that sufficient attention has not been directed to the careful control of subcritical temperatures and holding times. In many cases, rigid control of these variables is not emphasized. The erroneous reason advanced oftentimes to account for such laxity will be that "nothing happens anyway so why be so fussy?" The evidence presented by the authors proves that grain boundary precipitation at the higher quenching ranges and for extended holding times is the serious factor that is often ignored in the post mortem examination of failed tools. Even as the result of a routine metallographic survey, evidence of heavy grain boundary precipitation in many instances is not detected because if the tempered specimen under observation has been first etched for a length of time sufficient to fully develop the final structure, the outlines of the austenitic grains will be obliterated. We have found the use of a timed etch to be quite valuable in this respect. By etching specimens for brief periods in the order of 15-second intervals in 2 per cent nital, it is possible to gradually develop the final structure by repeated immersions and record observations of the grain boundary structures as revealed in the early stages of the etching procedure. It is significant to note that specimens showing evidence of heavy precipitation as disclosed by the short time etch will precisely match in final microstructure other specimens not characterized by this phenomenon when the etching is continued for a time sufficient to fully develop the final structure.

With respect to the effects of grain boundary precipitation promoted by some combination of times and temperatures used in hot quenching, the results of a series of transverse bend tests carried out by us at the Sanderson Works, Crucible Steel Company of America, Syracuse, New York, may be of interest.

[2]F. W. Taylor, "On the Art of Metal Cutting," an address published by The American Society of Mechanical Engineers. Third Edition, p. 226.

Table A
Schedule of Retarded Cooling Rate Experiments With 18-4-1 High Speed Steel

Heat	Carbon	Manganese	Silicon	Chromium	Vanadium	Tungsten
1-7567	0.73	0.32	0.24	4.11	1.11	18.33

Specimens ..... 0.540-Inch Rd.—8 Inches Long—Centerless Ground From ⅝-Inch Rd. Annealed Bars.

Initial Heat Treatment..... Preheated 1550 Degrees Fahr., Superheated 2350 Degrees Fahr., Soaked 1 Minute—Final Treatments Shown Below.

Group Number	Final Heat Treatment
1.	Oil-quenched to room temperature, tempered in salt bath for 1 hour at 1050 degrees Fahr., and air-cooled to room temperature.
2.	Placed directly in gas muffle furnace at 1200 degrees Fahr., for 3 hours, air-cooled to room temperature, tempered in salt bath for 1 hour at 1050 degrees Fahr., and air-cooled to room temperature.
3.	Quenched into lead bath for 5 minutes at 950 degrees Fahr., transferred directly to gas muffle furnace at 1200 degrees Fahr., for 3 hours, air-cooled to room temperature, tempered for 1 hour in salt bath at 1050 degrees Fahr., and air-cooled to room temperature.
4.	Placed directly in gas muffle furnace at 1200 degrees Fahr., held 1 hour, air-cooled to room temperature, tempered for 1 hour in salt bath at 1050 degrees Fahr., and air-cooled to room temperature.
5.	Quenched into lead bath for 5 minutes at 950 degrees Fahr., transferred directly to gas muffle furnace at 1200 degrees Fahr., for 1 hour, air-cooled to room temperature, tempered 1 hour in salt bath at 1050 degrees Fahr., and air-cooled to room temperature.
6.	Oil-quenched, removed from oil bath at approximately 1000 degrees Fahr., placed directly in salt bath at 1050 degrees Fahr., for 1 hour, and air-cooled to room temperature.
7.	Oil-quenched, removed from oil bath at approximately 1000 degrees Fahr., placed directly in salt bath at 1050 degrees Fahr., for 1 hour, air-cooled to room temperature, tempered at 1050 degrees Fahr., for 1 hour, in salt bath and air-cooled to room temperature.
8.	Oil-quenched, removed from oil bath at approximately 1000 degrees Fahr., cooled in still air for 15 minutes to approximately 230 degrees Fahr., placed directly in salt bath for 1 hour at 1050 degrees Fahr., and air-cooled to room temperature.
9.	Oil-quenched, removed from oil bath at approximately 1000 degrees Fahr., cooled in still air for 15 minutes to approximately 230 degrees Fahr., placed directly in salt bath for 1 hour at 1050 degrees Fahr., air-cooled to room temperature, tempered in salt bath for 1 hour at 1050 degrees Fahr., and air-cooled to room temperature.

In Table A are described the heat treatments applied to a series of 9 groups consisting of 6 specimens each produced from one electric arc heat of 18-4-1 high speed steel. The specimens were liberally ground before and after heat treatment in order to eliminate surface chemistry as a variable. Care was used to prevent the formation of grinding checks by use of a copious supply of coolant and by making the various removals by repeated steps. Five specimens of each group were subjected to transverse bend tests in a tensile machine while the sixth specimen was retained for microscopic study.

In this mechanical test, which we regard as a means of measuring the edge strength of a tool or cohesive strength of the outside fibers, smooth ground specimens free from scratches are required. Five specimens are tested per group and all results are used in determining the average values. The load is applied at a fairly rapid rate in order to simulate conditions under which many tools operate in service. Specifically, it is appreciated that minute edge crumbling or chipping can occur in many instances where the force causing failure of this type is rapidly applied but originates in intimate contact with the cutting edge of the tool. The results of these tests which are shown in Table B are calculated in terms of maximum fiber stress derived from the specimen size and

Table B
Transverse Bend Strength of 18-4-1 High Speed Steel as Influenced by Retarded Cooling Rates

Heat	Carbon	Manganese	Silicon	Chromium	Vanadium	Tungsten
1-7567	0.73	0.32	0.24	4.11	1.11	18.33

Specimens.....0.500-Inch Rd.—8 Inches Long—Centerless Ground From 0.540-Inch Rd. After Heat Treatment.
Span.....6 Inches Between Centers of Roller Supports.
Loading.....Applied Midway Between Supports at Rate of 6000 Pounds Per Minute.

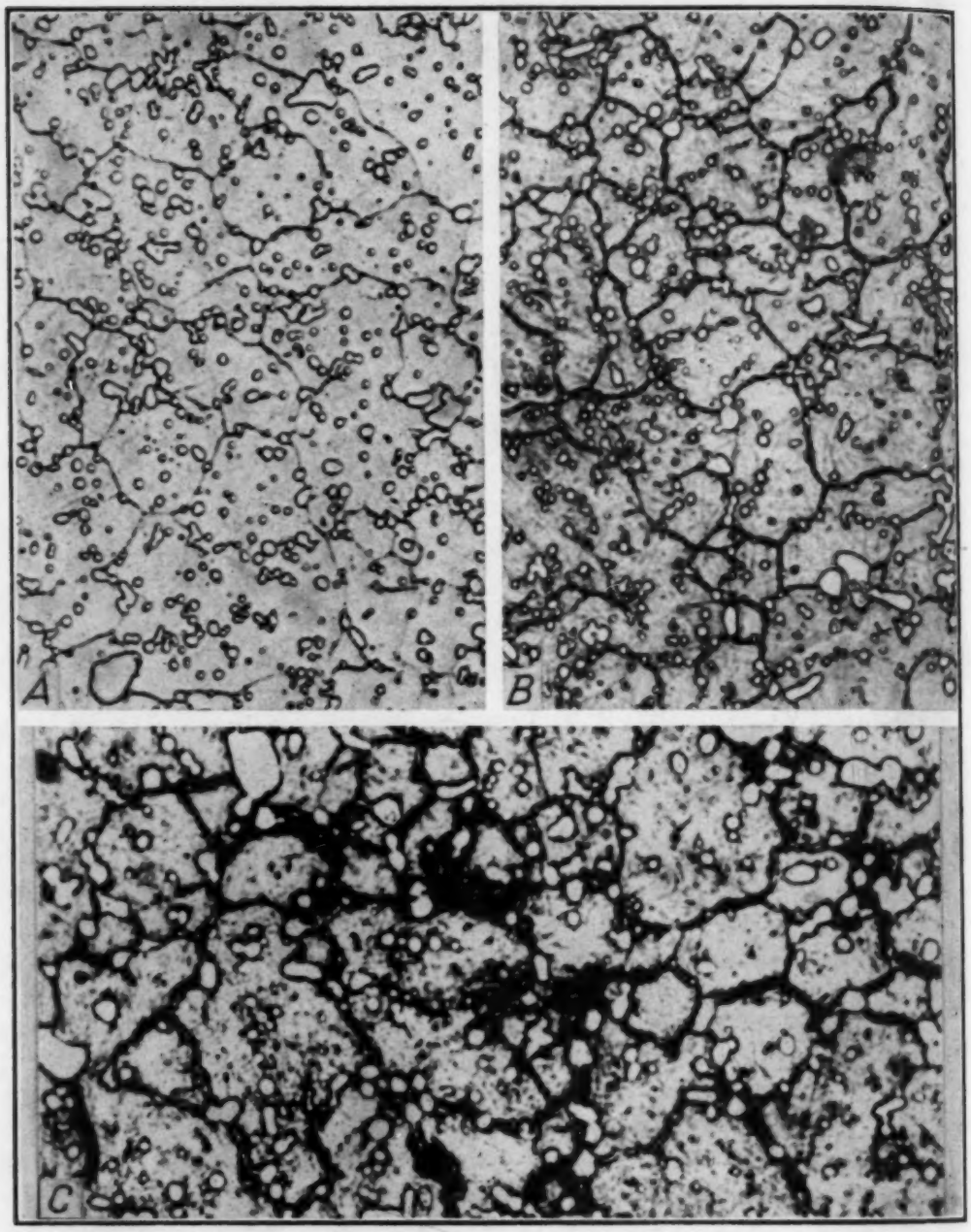
Group Number	Maximum Fiber Stress at Rupture* (1000 P.S.I.)	Extent of Experimental Error† Per Cent	Rockwell "C" Hardness	Description of Grain Boundary Structure (Etchant 2 Per Cent Nital—30 Seconds)
1	526	2	65.4	Grains Faintly and Incompletely Outlined.
2	362	3	61.8	Abundant Precipitation.
3	360	4	63.5	Abundant Precipitation.
4	422	3	64.0	Moderate Precipitation.
5	492	2	64.7	Fairly Moderate Precipitation.
6	94	52	66.0	Boundaries Not Resolved.
7	489	5	65.7	Fairly Moderate Precipitation.
8	444	4	65.7	Boundaries Not Resolved—Matrix Acicular.
9	524	2	65.3	Grains Faintly and Incompletely Outlined.

*Reported Values are Averages of 5 Specimens Per Group.
†Representing Deviation of Single Breaking Load Values With Respect to the Average Values for Each Group.

span used. The degree of experimental error shown beside the average fiber stress values is calculated by averaging the deviation from the mean of the 5 individual values. We prefer this method to the plan of discarding erratic individual values because it has been shown that some heat treatments are less consistent than others and for this reason the experimental error figures assume significant value. The results of treatment 6 illustrate this point. In this heat treatment involving no true tempering, the measuring error is so high that the average fiber stress value may be inconsistent and not reproducible.

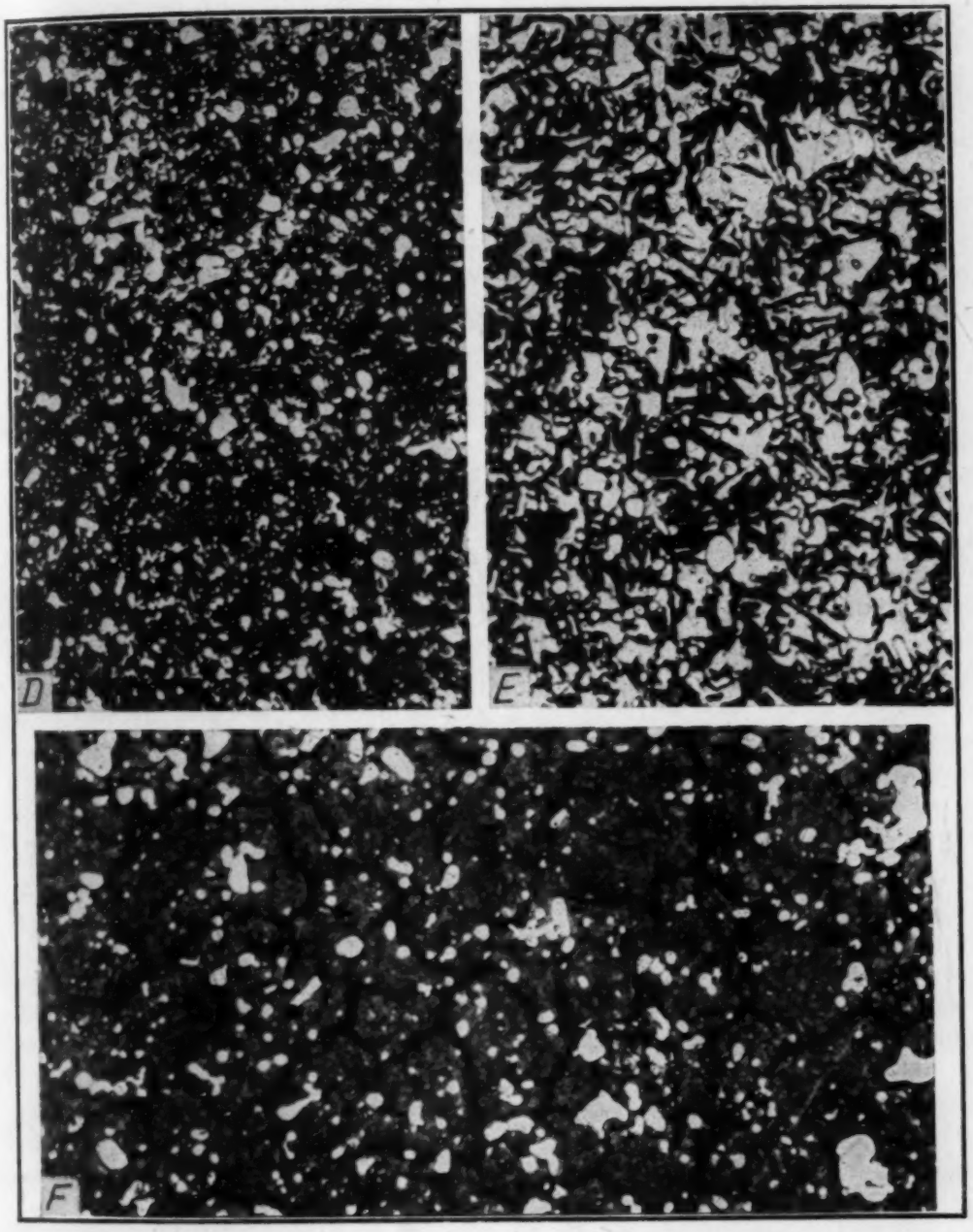
Treatment 3 will be recognized as similar to the practice recommended by Bornatsky.[3] As is evident from a comparison of the edge strength value with the grain boundary structure as revealed by the short time etch, there is a well defined correlation between strength value and degree of grain boundary precipitation. Fig. C illustrates the degree of grain boundary precipitate as revealed by the short time etch. At this arrested stage of etching, all of the grain boundaries have not been revealed. It is rather interesting to note in comparing the results of treatments 2 and 3, with treatment 1 as a standard, that the brief lead quench at 950 degrees Fahr. (510 degrees Cent.) had no apparent effect on the degree of precipitation when the quench was continued at 1200 degrees Fahr. (650 degrees Cent.) for 3 hours. However, when the time at 1200 degrees Fahr. (650 degrees Cent.) was limited to 1 hour, the brief immersion in lead at 950 degrees Fahr. (510 degrees Cent.) served to retard the degree of precipitation as shown by the results of treatments 4 and 5 respectively. The mechanical test results of treatments 5 and 7 are only slightly less than

[3]I. Bornatsky, "Improving the Resistance of High Speed Tools," *Stal*, 1939, No. 6, p. 44-46.



Figs. A-C—Effects of Timed Etching on Grain Boundaries of 18-4-1 High Speed Steel. Superheated 2350 Degrees Fahr. for 1 Minute. 2 Per Cent Nital Etch. × 1000. Fig. A—Oil-Quenched to Room Temperature. Etched 180 Seconds. Fig. B—Oil-Quenched, Removed from Oil Bath at Approximately 1000 Degrees Fahr., Placed Directly in Salt Bath at 1050 Degrees Fahr., Held 1 Hour and Air-Cooled to Room Temperature. Etched 180 Seconds. Fig. C—Quenched Into Lead for 5 Minutes at 950 Degrees Fahr., Placed Directly in Gas Muffle Furnace at 1200 Degrees Fahr., Held 3 Hours, Air-Cooled to Room Temperature, Tempered 1050 Degrees Fahr., Held 1 Hour and Air-Cooled to Room Temperature. Etched 30 Seconds.

that of standard treatment 1. It is questionable, therefore, whether such treatments would seriously affect the edge strength of tools in service. The assembly of test data derived from various sources would be of substantial value in determining the amount of precipitation that can safely be permitted.



Figs. D-F—Microstructures of 18-4-1 High Speed Steel. × 1000. Superheated 2350 Degrees Fahr., for 1 Minute. Fig. D—Oil-Quenched to Room Temperature, Tempered 1 Hour in Salt Bath at 1050 Degrees Fahr., and Air-Cooled to Room Temperature. Etched in 2 Per Cent Nital for 120 Seconds. Fig. E—Oil-Quenched, Removed from Bath at Approximately 1000 Degrees Fahr., Air-Cooled to Approximately 230 Degrees Fahr., Placed Directly in Salt Bath at 1050 Degrees Fahr., Held 1 Hour and Air-Cooled to Room Temperature. Etched in 2 Per Cent Nital for 120 Seconds. Fig. F—Oil-Quenched to Room Temperature, Tempered at 1050 Degrees Fahr., Held 1 Hour and Air-Cooled to Room Temperature. Etched in Watson Reagent (5 Per Cent Boiling Aqueous Picric Acid Solution) for 150 Seconds.

The results of treatment 8 which duplicates heat treatments thoroughly discussed by Palmer[4] show how the edge strength can be lowered by not completing the quench prior to tempering. After proper tempering, as shown by results

[4]Frank R. Palmer, "Tool Steel Simplified," published by The Carpenter Steel Company, Reading, Pennsylvania, 1937, p. 285, 286.

of treatment 9, the values duplicate those of standard treatment 1. As Palmer has emphasized, the avoidance of such structures as depicted by Fig. E involves merely an application of good heat treating principles but such structures are frequently found on tools examined from time to time. It can be perhaps visualized that since the calculated edge strength of treatment 8 is only approximately 15 per cent less than that of standard treatment 1, tools so treated could "get by" with mediocre performance in some instances. Practical observations have proved that such structures have a detrimental effect on tool performance and in doubtful cases where it is not convenient to make a metallographic examination, thereby destroying the tool, it is always advisable to repeat the tempering operation. The microstructure depicted by Fig. D is representative of both treatment 1 and 9.

In establishing proper laboratory technique for the examination of heat treated samples, it is highly advisable to be provided with a series of "standards" heat treated under a variety of known conditions. The samples under observation should then be prepared for metallographic study together with the standards. It is also recommended that a master stock nital solution be used for time etch examination. By preparing the "unknowns" along with the standard specimens, variables such as oxide films and temperature of the etching solution can be contemplated and corrected because such factors have an important influence on the rate of etching. The final structures of properly tempered samples of 18-4-1 high speed steel are usually fully developed at the end of a total time of 2 minutes in 2 per cent nital. Samples oil quenched to room temperature but not tempered require longer times. Fig. A shows the microstructure of an underetched specimen but illustrates the grain boundary effect when compared to Fig. B, representative of a specimen etched for the same time.

At the completion of the structural examination by means of the nital etch, grain counts by the Snyder-Graff intercept method[5] can be made. For specimen preparation, it is recommended that the prior etch be removed by suitable polishing and the specimens be then immersed in a freshly prepared boiling aqueous solution of 5 per cent picric acid for a period of 2 to 3 minutes. This solution has been found to be particularly valuable in the routine examination of larger numbers of specimens. Repeated polishings and etchings are not necessary and their use facilitates the making of grain counts even on specimens of 18-4-1 high speed steel heated to superheating temperatures as low as 2200 degrees Fahr. (1095 degrees Cent.). Because of its explosive nature when evaporated, care must be exercised to completely dispose of the solution after use. It must likewise be employed with caution when studying grossly overheated samples. Owing to its deep etching properties, cracks are developed within the grains of such specimens and may sometimes be confused with grain boundaries. It is for this reason that we recommend use of the special etch after the microstructural examination as determined by the nital etch is first completed.

Written Discussion: By W. E. Bancroft, chief metallurgist, Pratt & Whitney Division, Niles-Bement-Pond Co., Hartford, Conn.

The importance of this splendid paper will probably not be fully realized in

[5]R. W. Snyder and H. F. Graff, "Study of Grain Size in Hardened High Speed Steel," METAL PROGRESS, April 1938, p. 377.

a practical sense until a thorough correlation has been established between the material contained therein, and the hardness and physical properties of high speed steel when quenched under different conditions. This the authors have indicated they are preparing to do. It seems necessary now to determine what constitutes the optimum condition or combination of transformation products before tempering in order to produce the most serviceable tools. Once this has been established, the information in this paper should certainly provide an adequate and practical method of controlling conditions so as to produce consistently the types of structure most to be desired.

It is true that for years it has been the practice of some manufacturers to quench high speed steel tools in liquid baths between 1000 and 1200 degrees Fahr. (540 and 650 degrees Cent.). Probably, in most cases, the actual holding time in the quench has not been long enough to permit isothermal transformation but many operators have felt it highly desirable to cool slowly from the quench to room temperature. Experience has seemed to show that this reduced the tendency to crack certain poorly proportioned tools. Undoubtedly, considerable bainite has been formed in this way and not recognized as such. As the authors state, it is not yet known whether the presence of bainite before tempering is detrimental or not. Practical experience would indicate that it may not be particularly harmful—certainly thousands of tools so treated have given satisfactory service in the field. However, it will be very interesting to find out more about this, using the principles developed in this paper as a means of producing controlled results.

Another practice which can well be investigated, in the light of the information presented in this paper, is that of "Hot Straightening". Here we have a variable condition—different tools and in most cases different portions of the same tool being held at elevated temperature for varying lengths of time, also the rate of cooling from the quench to room temperature must vary considerably. Possibly, this variation may account for some of the hitherto unexplainable behavior of certain individual high speed steel tools which sometimes give outstandingly good or poor service in the field.

Written Discussion: By W. R. Frazer, metallurgist, Union Twist Drill Co., Athol, Mass.

We are again indebted to Dr. Cohen and his associates for more information on the heat treatment of high speed steel and we wish to congratulate them on the thorough manner in which they have studied the transformation of austenite by means of hot quenching.

The transformation curves drawn by the authors in Figs. 15 and 16 are similar in shape but differ in location to those developed by Payson and Klein as reported in their current paper entitled "The Hardening of Tool Steels", and also those shown in the literature covered by items 7 and 8 cited in the bibliography. However, we would like to call attention to the knee of the upper curves, all of which occur between 1320 and 1400 degrees Fahr. (715 and 760 degrees Cent.) and suggest that hot quenching should be done below these temperatures to prevent any isothermal reaction. It has been shown by Ham, Parke and Herzig that the isothermal product in this zone is a soft constituent occurring first at the austenite grain boundaries and, if time is allowed, the

entire grain will be transformed to a soft condition. It is our opinion that 1200 degrees Fahr. (650 degrees Cent.) is the maximum temperature that should be used for hot quenching of high speed steel.

We are sorry that the authors did not include the hardness values resulting from the various treatments, particularly in the range covered by the lower family of C curves. Gulyaev (8) shows a decrease in hardness from Rockwell C-66.0 to C-53.0 by isothermal decomposition at 592 degrees Fahr. for 30 minutes for 15 per cent tungsten, 3 per cent chromium steel. Ham and associates (7) show a decrease from Rockwell C-65.0 to C-55.0 at 600 degrees Fahr. in 225 minutes for 6-6-2 high speed steel. Similar data by the authors would be of value. However, the above figures indicate that the formation of bainite above the M-point definitely lowers the hardness, so it is doubtful if isothermal treatment above the M-point is desirable for cutting tools where high hardness is required.

If we consider the curves below the M-point, it is evident that this paper, together with that by Payson and Klein, confirms the work of Carpenter and Robertson (16) also Greninger and Troiano (13) that austenite transforms to martensite only as a result of decreasing the temperature and the amount of martensite formed is related to the lowering of the temperature. Figs. 3, 5, and 17 show that although the amount of martensite formed is a constant at any given temperature below the M-point, isothermal reactions will take place with the formation of bainite. We would be interested in the hardness of this product, also the results of transverse bend tests to determine the toughness of this structure as compared with the results reported by two of the authors last year[6] and, of course, we are all anxious to study the next chapter of this investigation; namely, what is the effect of tempering on these isothermal products, their hardness, toughness, etc.

Written Discussion: By J. L. Klein and P. Payson, Eastern Research Laboratory, Crucible Steel Company of America, Harrison, N. J.

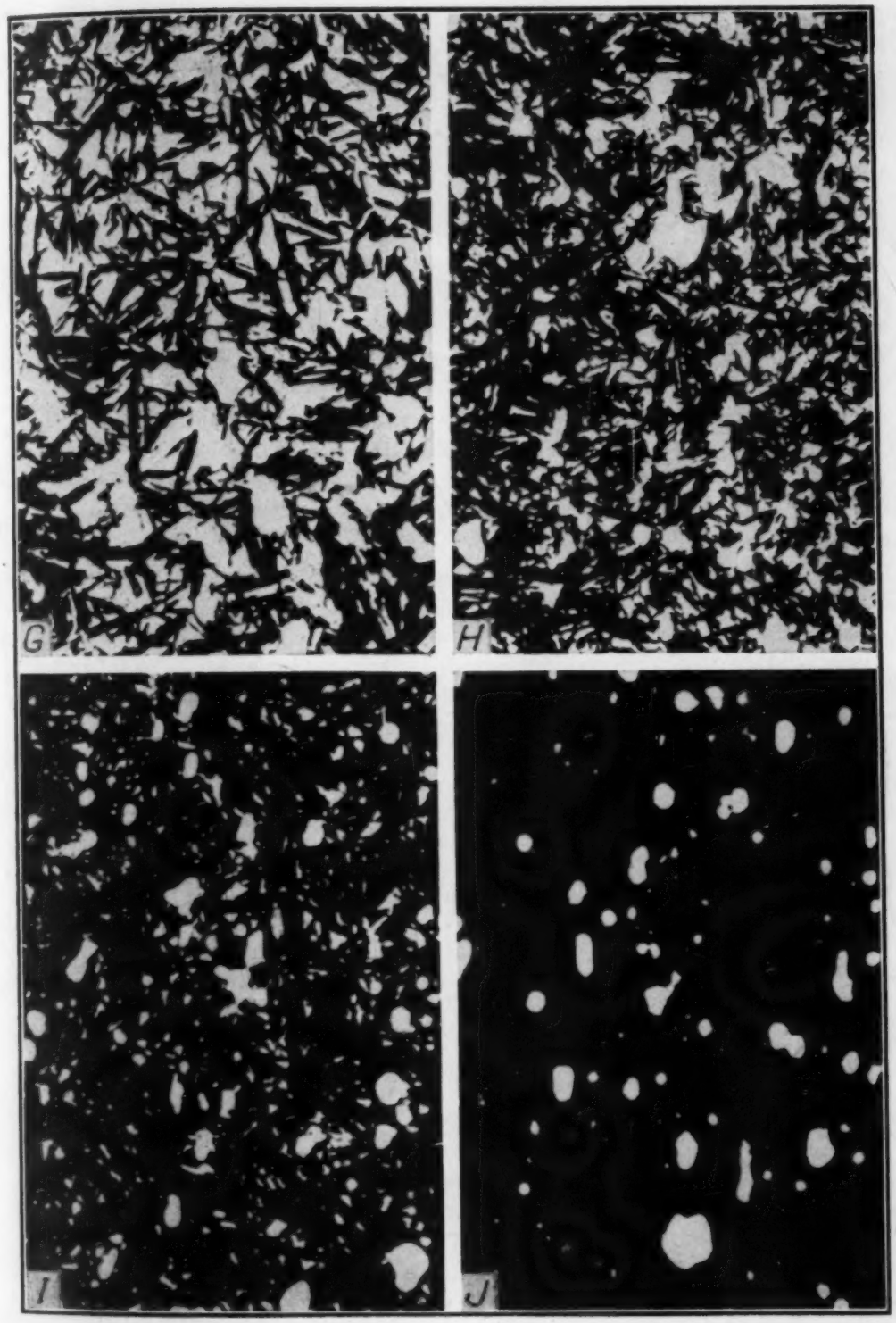
The data presented by the authors for the transformation of austenite in high speed steels are, generally, in fairly good agreement with the results determined independently in our own laboratory, and presented in another paper at this session.[7] However, there are some differences which seem worthy of discussion. Dr. Cohen generously supplied us with some of the stock used to obtain the data for this paper so that any differences in his results and our results could not be attributable to differences in composition, or melting practice.

The authors have referred to the transformation product formed at high temperatures (down to 1200 degrees Fahr.) as "pearlite" and "fine pearlite". The structure is spheroidal rather than lamellar and we prefer to use the name "fine spheroidite" rather than "fine pearlite" for the product formed at these temperatures.

According to the transformation curves of Figs. 15 and 16 of this paper, the 6-6-2 and 18-4-1 high speed steels which have been cooled rapidly enough

[6]Paul Gordon and Morris Cohen, "The Transformation of Retained Austenite in High Speed Steel at Subatmospheric Temperatures," TRANSACTIONS, American Society for Metals, Vol. 30, 1942, p. 569-591.

[7]P. Payson and J. L. Klein, "The Hardening of Tool Steels," published in this issue of TRANSACTIONS, American Society for Metals, p. 218.



Figs. G-J—Retention of Austenite in 6-6-2 High Speed Steel. Specimens Austenitized at 2235 Degrees Fahr. Etched in 10 Per Cent Nital. × 1000. Fig. G—Cooled to 200 Degrees Fahr. 41 Per Cent Untransformed Austenite. Fig. H—Cooled to 150 Degrees Fahr. 25 Per Cent Untransformed Austenite. Fig. I—Cooled to 100 Degrees Fahr. 12 Per Cent Untransformed Austenite. Fig. J—Cooled to Room Temperature and Double Tempered. 9 Per Cent Carbides.

from the austenitizing temperatures, so that they transform only to martensite, have considerable amounts of austenite untransformed, or "retained", at various low temperatures. We have used the Greninger-Troiano technique to determine the amounts of martensite at low temperatures in the two high speed steels furnished by Dr. Cohen. The 6-6-2, austenitized at 2235 degrees Fahr. (1225 degrees Cent.), and the 18-4-1, austenitized at 2350 degrees Fahr. (1290 degrees Cent.), were quenched to 200, 150, and 100 degrees Fahr. (95, 65, and 40 degrees Cent.), respectively, and tempered immediately at 1100 degrees Fahr. (595 degrees Cent.). Control samples, quenched to room temperature and double tempered, so that everything except the carbides undissolved by the austenitizing treatment, etched dark in nital, were used to determine the volume of the steel occupied by the undissolved carbides. Figs. G to J and K to N represent the structures obtained for the 6-6-2 and 18-4-1, respectively. The dark-etching products represent the austenite that has transformed to martensite on the quench to the low temperature (200, 150 or 100 degrees Fahr.), and the white etching product represents a mixture of austenite untransformed at the low temperature, plus carbides undissolved at the austenitizing temperature. To measure the amounts of transformation, the point counting technique described by the authors was used. Since it is difficult to distinguish the undissolved carbides from the remainder of the white etching portions of the photomicrographs, the total white area for each sample was determined, and from it was subtracted the amount of carbides found in the control samples (Figs. J and N).

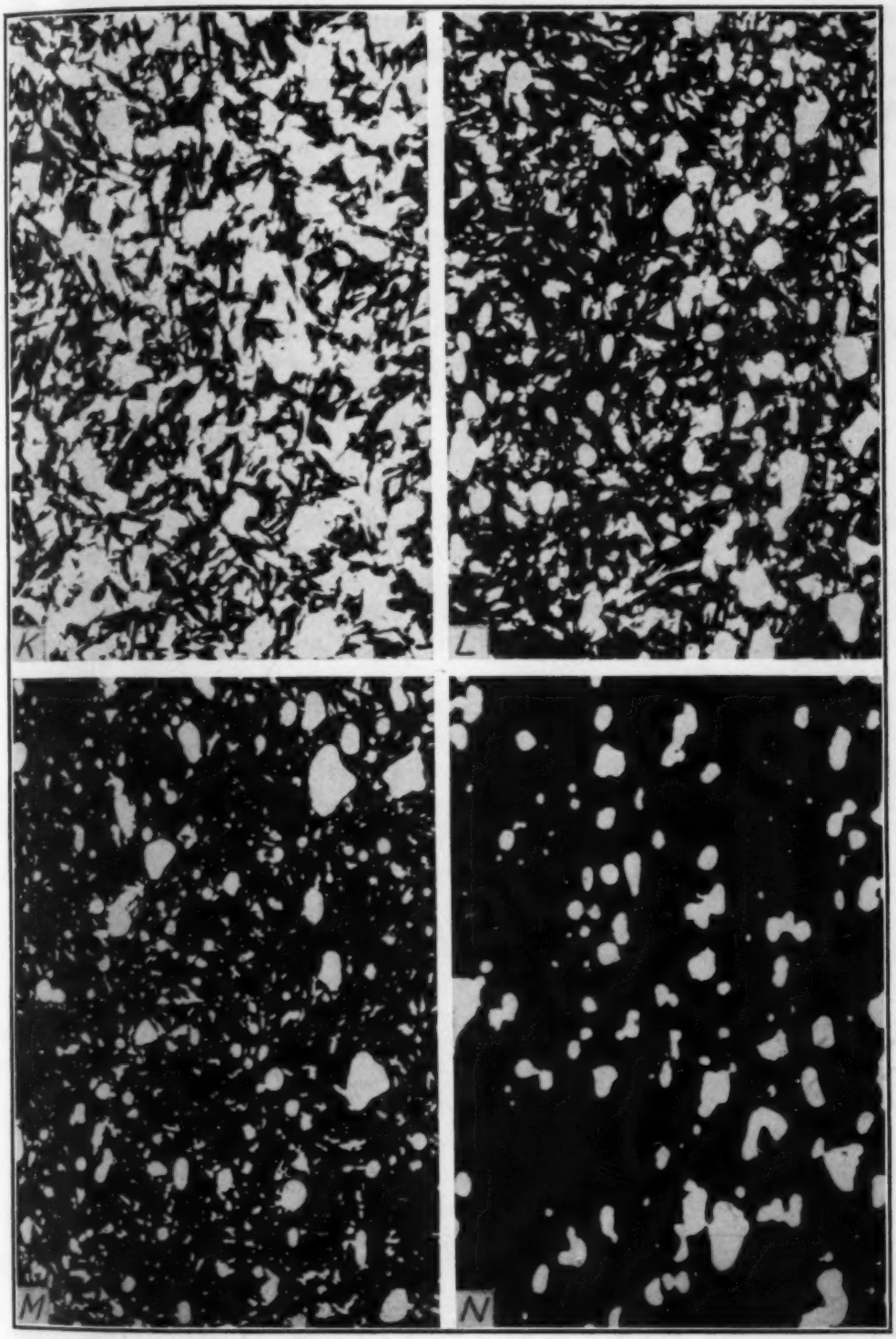
The fields shown represent longitudinal sections which show carbide streaks. These fields were chosen for measurements as they represent the maximum amounts of retained austenite to be found in the steels. The control samples (Figs. J and N) show carbide-rich streaks which correspond to those of the alloy-rich streaks shown in Figs. G to I and K to M.

A comparison of the results found by Dr. Cohen and his associates by the dilatometric method, and by us by the metallographic method, is given below:

	Percentages of Untransformed Austenite in			
	6-6-2 Austenitized at 2235 Degrees Fahr.		18-4-1 Austenitized at 2350 Degrees Fahr.	
At	Dilatometric Method of Authors	Metallographic Method of Discussers	Dilatometric Method of Authors	Metallographic Method of Discussers
200° F.	50	41	40	34
150° F.	40	25	*33	19
100° F.	30	12	*27	7

*Interpolated from values given in Fig. 16.

In addition to the differences between the authors' estimate of untransformed austenite as established by the dilatometric technique, and our estimate as established by the metallographic technique, there appear also to be differences in the respective estimates when both use the metallographic technique. In Fig. 15, the authors state that the average amount of untransformed austenite in 6-6-2 at 200 degrees Fahr. (95 degrees Cent.), as determined by their metallographic technique, is 53 per cent. Our determination of the maximum amount of untransformed austenite in this same steel at 200 degrees Fahr. (95 degrees Cent.) is 41 per cent. In their metallographic technique, the authors use a tempering of only 5 seconds at 1050 degrees Fahr. (565 degrees Cent.).



Figs. K-N—Retention of Austenite in 18-4-1 High Speed Steel. Specimens Austenitized at 2350 Degrees Fahr. Etched in 10 Per Cent Nital. × 1000. Fig. K—Cooled to 200 Degrees Fahr. 34 Per Cent Untransformed Austenite. Fig. L—Cooled to 150 Degrees Fahr. 19 Per Cent Untransformed Austenite. Fig. M—Cooled to 100 Degrees Fahr. 7 Per Cent Untransformed Austenite. Fig. N—Cooled to Room Temperature and Double Tempered. 15 Per Cent Carbides.

We used a tempering of 10 minutes at 1100 degrees Fahr. (595 degrees Cent.). We have found in our quench-temper procedure on a wide variety of steels that martensite tempered for very short times may appear light etching unless very long etching times are used. It would be interesting to know how long an etching time the authors used for the sections which were photographed for Fig. 17. The sections which we photographed for this discussion were etched for 2 minutes in 10 per cent nital.

Unfortunately, we can offer no satisfactory explanation for the discrepancy between the authors' results based on the extrapolations from the dilatometric measurements, and our results based on the metallographic procedure. We are sure that some carbides precipitated from the austenite in the dilatometer samples because they were air-cooled down to the martensite point. However, this precipitation would lower the carbon content of the austenite, and therefore decrease the amount of retained austenite, rather than increase it.

We should also conclude from the hardness measurements that there is less than 25 or 30 per cent retained austenite in a quenched sample. The hardness of 18-4-1, for example, is about 65 Rockwell "C" as-quenched, and rises to only 65.5 or 66 Rockwell "C" after a 2-hour temper at 1050 degrees Fahr. (565 degrees Cent.), which is sufficient to "condition" substantially all of the retained austenite. After several multiple 2-hour tempers at 1050 degrees Fahr. (565 degrees Cent.), when the hardness effects are attributable only to the tempering of martensite, the hardness is still 64 to 65 Rockwell "C". We have considerable data on many high alloy steels containing 0.80 to 0.95 per cent carbon in which the difference in hardness between the steel as-quenched and after adequate tempering is as much as 10 to 11 points Rockwell "C". For example, one steel which we estimated contained about 30 per cent untransformed austenite before it was tempered, had a hardness of C-57 as-quenched and C-65 after sufficient tempering at 1050 degrees Fahr. (565 degrees Cent.) to eliminate all retained austenite.

One other point, although qualitative in nature, is worthy of note. For about 25 years or more, 18-4-1 has been used very successfully as a general purpose steel for cutting tools. For most of that time only a single temper was used after the quench. If the authors' calculations of the amounts of retained austenite are correct, then these tools must have contained about 20 to 30 per cent untempered martensite when they were put into service. Although a steel containing that much untempered martensite may do a good cutting job when it is firmly supported and making a smooth cut, it is hard to believe that tools which require some toughness could give satisfactory service with as much as 20 to 30 per cent untempered martensite in them.

We should like to know if the authors have an explanation for the extremely interesting phenomenon which occurs when high speed steel is hot-quenched to temperatures between 600 and 400 degrees Fahr. (315 and 205 degrees Cent.), and held at these temperatures for some time. We have also found that the untransformed austenite is stabilized by such a treatment so that it does not transform during subsequent cooling to room temperature, and we have encountered the same behavior in alloy steels other than high speed steel.

Oral Discussion

O. E. HARDER:[8] There are two suggestions which may be of interest in connection with this type of work. First, in connection with the study of the balance in the composition of 25 per cent chromium, 12 per cent nickel alloys, as reported last year by Gow and Harder, we and the Leeds and Northrup Company have developed equipment for magnetic permeability testing to determine low concentrations of ferrite. That was important in connection with that alloy. The first unit of that equipment is supposed to be available shortly and it might have some application in studying the amount of retained austenite in these steels. However, our alloys are predominantly austenitic while high speed steels are probably highly ferritic and some modification of the equipment might be necessary.

The second suggestion relates to the hardness of these materials at temperature. For example, it is apparently possible to use an arrested hot quench of these steels and then by using equipment like what was described by Harder and Grove[9] a few years ago, you could determine the hardness at this temperature. You could determine the hardness of the material after holding the temperature for a while. This would make it possible to take specimens in that range in which you get bainite and hold them at that temperature and study the hardness over a period of time. That would give some information on the hardness at temperature. Also this method might give some valuable information on whether or not these steels are materially hardened on reheating or if the hardening, as is now generally assumed, all takes place on the further cooling from the tempering temperature to room temperature. Such data would give some definite information along that line.

Authors' Reply

The authors wish to express their appreciation for the considerable interest in this paper shown by the many discussers, and in particular wish to thank Professor Trigger and Messrs. Weldon and Watson for their valuable additions to the subject investigated.

Professor Trigger's data on the hot quenching of 6-6-2 steel at 1025 degrees Fahr. (550 degrees Cent.) indicate very good agreement with ours. However, the values representing our data in his second table are incorrectly chosen in some cases, and when the corrections are made (as shown in Table C below) the agreement is even better. Our values for the beginnings of transformations are obtained from dilatometric curves and are taken as the first noticeable break in slope of the normal cooling curve. Similarly our peak values (i.e., the temperatures at which the maximum austenite transformation rates occur) have been chosen at the points of maximum slope in the upward swing of the dilation curves. The chief discrepancy between the two sets of figures occurs in the bainite inception temperatures after 2 and 24 hours of hot quenching. The explanation for this may lie in the fact that the hot quenching temperature

[8]Assistant director, Battelle Memorial Institute, Columbus, Ohio.

[9]Harder and Grove, *Transactions*, Iron and Steel Division, American Institute of Mining and Metallurgical Engineers, Vol. 105, 1933, p. 88-124.

(1025 degrees Fahr.) used by Professor Trigger was somewhat higher than that (1000 degrees Fahr.) for which our data are given.

Table C
Comparison of Austenite Transformation Temperatures

Holding Time Hours	Transformation Temperatures, Degrees Fahr. Gordon, Cohen, and Rose — Bainite Begin	Bainite Peak	Martensite Begin	Martensite Peak	Trigger — Bainite Begin	Bainite Peak	Martensite Begin	Martensite Peak
0	...	...	420	320	...	...	370	290
2	500	...	400	340	570	510	390	330
24	580	530	330	290	640	540	310	290

Dr. Butler asks whether we consider the temperature range of 700 to 900 degrees Fahr. (370 to 480 degrees Cent.) as the best for hot quenching. The answer to this question depends upon the purpose of the hot quenching operation. (1) The answer is "yes" if the aim of the hot quench is merely the reduction of temperature gradients or the facilitation of cleaning operations with the intention of obtaining a normal hardened structure. The reason for this is simply that hot quenching in this range produces no detectable austenite decomposition at temperature and exerts little or no influence on the transformation during subsequent cooling. (2) If, on the other hand, it should be ascertained that certain combinations of martensite, bainite, and austenite are definitely beneficial, and if the purpose of the hot quench is to produce such structures, then hot quenching in the range of 700 to 900 degrees Fahr. (370 to 480 degrees Cent.) will undoubtedly prove ineffectual, as stated in conclusion 5 of the paper.

The observations made by Dr. Butler in the last paragraph of his discussion require some modification. First, hot quenching to 500 degrees Fahr. (260 degrees Cent.) for any appreciable length of time will result in considerable quantities of bainite approaching 30 per cent of the total structure if the holding time is as little as 2 hours, and 55 to 60 per cent if the time is 24 hours. Second, the amount of retained austenite in the steel at room temperature after such hot quenching is appreciably greater than in the case of normally quenched steel. After a 24-hour hot quench at 500 degrees Fahr. (260 degrees Cent.), for example, the amount of residual austenite in the hardened steel at atmospheric temperature is in the neighborhood of 40 to 45 per cent of the total structure. This is approximately twice the amount of austenite in the steel after a direct quench. It is evident, therefore, that during subsequent tempering there would be more retained austenite to transform in the hot quenched steel than in the directly quenched steel, and there would be correspondingly more expansion during that transformation.

The excellent data presented by the Messrs. Weldon and Watson are particularly welcome because they support the authors' contention that deleterious effects may result from the standard practice of hot quenching high speed steel if the bath temperature is too high. They have independently checked our observations that grain boundary carbide precipitation occurs during hot quenching at temperatures over about 1200 degrees Fahr. (650 degrees Cent.) and have added real evidence that such precipitation either in itself or by its influence on the products of austenite transformation has an adverse effect on

the strength of the steel. It must not be assumed, however, that this carbide precipitation has detrimental effects on all the mechanical properties of the steel for, as Bornatsky has indicated, such precipitation may be accompanied by a large increase in lathe tool life. This would not be the first instance in which a treatment productive of inferior toughness was at the same time conducive to improved single edge cutting tool efficiency.

The etching solutions and technique described by Messrs. Watson and Weldon appear to be very useful not only in detecting the presence of grain boundary precipitation, but in indelibly delineating the austenitic grain size. Their Figs. A and C show in excellent contrast the effect of grain boundary precipitation. If the etching time of the two specimens had been identical, the contrast would undoubtedly have been more marked.

The remarks by Mr. Bancroft and Dr. Frazer prompt us to present some tentative hardness values we have obtained on hot quenched 6-6-2 steel. These values are given in Table D and are to be regarded merely as preliminary indications of the hardness trends and subject to change upon more complete investigation.

Table D
Hardness Values of Hot Quenched 6-6-2 Steel

Hardening Temperature—2235 Degrees Fahr.; Untempered
Cooling Rate from Hot Quenching Treatment
5-10 Degrees Fahr. Per Minute

Hot Quenching Temperature Degrees Fahr.	Hot Quenching Time, Hours	Rockwell "C" Hardness
80	..	66.0
212	24	66.2
300	2.5	65.7
300	24	64.5
350	24	63.2
400	24	57.5
500	24	57.1
600	0.1	66.0
600	0.5	59.5
600	2.0	54.2
600	24	52.8
700	24	65.9
700	165	65.9
800	24	66.0
900	24	66.2
1000	2	65.9
1000	6	66.3
1000	24	65.9
1100	0.25	64.3
1100	0.5	64.2
1100	24	63.8
1200	24	57.9
1230	24	46.5
1300	24	34.1
1350	24	26.3

Dr. Harder's suggestions are appropriate. We appreciate the sensitivity of the magnetic method and, as a matter of fact, we are now using just such a method along with the dilatometric and other techniques for following the course of austenite decomposition. Also, a hot hardness tester, built on the Rockwell principle, has been used at Massachusetts Institute of Technology to follow isothermal transformations. A description of the apparatus and some

typical results have been published recently.[10] We expect to investigate the hot hardness characteristics of hot quenched structures in the near future.

Messrs. Payson and Klein rightly point out that the product of isothermal transformation in the upper range of austenite instability is not lamellar in these steels. As mentioned on page 187 of the paper we have used the term pearlite in a loose sense. When the temperature and extent of the austenite decomposition are such as to permit resolution of the aggregate, the carbides appear spheroidal rather than plate-like.

We are glad to know that Messrs. Payson and Klein check our results relating to the stabilizing effect of bainite formation on the austenite which remains untransformed. This phenomenon is apparently a rather general one, and any theory of bainite formation will certainly have to take it into account. At this time we can offer no tenable explanation for this stabilizing effect.

It is our belief that the estimates of the amounts of retained austenite in 6-6-2 and 18-4-1 high speed steels at room temperature after direct quenching as given by Messrs. Payson and Klein are exceedingly low. The following arguments are presented in substantiation of this belief. (1) Messrs. Payson and Klein have used longitudinal sections of the steels for their metallographic examinations. Such sections are in general not representative of the volume of the steel because there is considerable axial segregation of the carbides. Whereas any transverse section, if properly sampled, will give a fairly representative picture of the carbide distribution throughout the steel volume, the amount present on any one longitudinal section will vary with the distance of that section from the bar axis. We have found in sample after sample that the average amount of undissolved carbide measured on transverse sections is 5 to 7 per cent for the 18-4-1 and 5 to 6 per cent for the 6-6-2 used in our investigation. Messrs. Payson and Klein report 15 per cent carbide for the 18-4-1 and 9 per cent for the 6-6-2. Since the amount of carbide must be subtracted from the total white constituent to obtain the austenite content, it is obvious that high carbide determinations will lead to low austenite values. This does not eliminate the entire discrepancy between their findings and ours but does at least close part of the gap. (2) The metallographic method is not applicable to all steels, nor even to all structures of a given steel, with the same degree of accuracy. When the austenite present is not large in amount and when it is intimately mixed with the martensite, the metallographic method is out of line with the magnetic, specific volume, dilatometric, and X-ray methods which we have employed for measuring per cents of austenite. With such mixtures of austenite and martensite, it becomes very easy to obscure part of the austenite by over tempering, over etching, or improper photographing conditions. The structure obtained in directly quenched high speed steel is just such a nondescript mixture. At hot-quenching temperatures of 200 degrees Fahr. (95 degrees Cent.) and above, however, where the austenite is present in a condition which lends itself readily to measurement by the metallographic method, the estimates of per cent austenite made by Messrs. Payson and Klein and those made by us are in fairly good agreement. (3) The increase in specific volume

[10] E. C. Bishop and M. Cohen, "Hot Hardness Tests of High Speed Steel with Rockwell Tester," METAL PROGRESS, Vol. 43, March 1943.

attendant to the tempering of 18-4-1 and 6-6-2 steels can be associated only with the transformation of residual austenite. We have measured this increase in specific volume very accurately and have found that for 18-4-1 it is 0.00045 cc/gm. If this enlargement is the result of the decomposition of only 2 per cent austenite (this is the amount indicated by Payson and Klein for 18-4-1 at 80 degrees Fahr.)[11] then complete transformation, i.e., the change from the primary austenitic to the quenched and fully tempered state, would effect an expansion of 0.0225 cc/gm or about 21 per cent. S. G. Fletcher[12] and A. B. Greninger,[13] however, have independently estimated the change in volume accompanying the gamma-alpha transformation in straight carbon steel as 3.5 to 4 per cent. Our own measurements indicate that in high speed steel this value is probably closer to 3 per cent. It is evident from these figures that the increase in volume on the basis of 2 per cent retained austenite is about 7 times the calculated amount. We doubt that many high speed steels could accommodate themselves to a 21 per cent increase in volume during the hardening operation. (4) Messrs. Payson and Klein have indicated the use of hardness measurements to check their per cents of retained austenite. It was shown very nicely in the paper by Dr. Roberts and Dr. Mehl[14] that in a structure containing as much as 15 per cent pearlite and 85 per cent martensite the hardness values would not reveal the presence of pearlite. Furthermore, we have produced as much as 20 per cent retained austenite in a 1.00 plain carbon steel and found the hardness to be R_c 64, and 40 per cent retained austenite in a 1.25 plain carbon steel and measured the hardness as R_c 60. Hence, despite large amounts of austenite present in the structure the hardness values may be very high and in themselves not indicate quantitatively the presence of the austenite.

Long before there was any systematic study of the tempering process in high speed steel, many small tool manufacturers were giving fine and multiple edge tools a "double temper". This extra operation had been well warranted by the observation that tools so treated had superior toughness, although it is doubtful whether maximum benefits were achieved due to an insufficient understanding of the time factors involved. If only 2 per cent of the structure was austenite (untempered martensite after a single temper) then the superiority observed after multiple tempering was solely the result of an effect on this 2 per cent. We feel that the magnitude of the improvement in static and impact properties is more consistent with the modification of a much greater portion of the structure.

Mr. Payson doubts that a high speed tool containing 20 to 30 per cent untempered martensite would give satisfactory service for an application requiring some toughness. "Satisfactory service" and "some toughness" are difficult terms to interpret, since they are both subject to almost continuous revision. However, as has been indicated, better results were in fact obtained on applications requiring a high order of toughness when the percentage of untempered martensite was reduced through the expedient of multiple tempering.

---

[11]P. Payson and J. L. Klein, "The Hardening of Tool Steels," published in this issue of TRANSACTIONS, American Society for Metals, p. 218.

[12, 13]Personal Communication.

[14]G. A. Roberts and R. F. Mehl, "The Mechanism and the Rate of Formation of Austenite from Ferrite-Cementite Aggregates," American Society for Metals Preprint 22, 1942.

THE HARDENING OF TOOL STEELS

By P. Payson and J. L. Klein

Abstract

The reactions which take place during the heat treatment of eleven commercial tool steels, from plain carbon to high speed, have been studied, and the transformation temperature time curves of the austenites of these steels are presented. The formation of martensite in these steels is discussed, and emphasis is placed on the need for cooling the steels to low temperatures, in the hardening operation, before tempering is started. Finally, some data are presented to show the effects in these steels of variations in austenitizing temperatures on the formation of martensite, and on the retention of austenite at room temperature.

Introduction

THE S-curve—or, as the authors prefer to designate it, the Transformation Temperature Time, or TTT, curve—has helped, beyond measure, to explain some of the reactions which may occur in steel when it is heat treated. Up to the present time, very few curves have been published on commercial tool steels, and few of these, in the opinion of the authors, adequately indicate the reactions which occur when austenite transforms at temperatures below about 500 degrees Fahr. (260 degrees Cent.). Because of the need for more information on the transformation of austenite in tool steels, the personnel of the laboratory with which the authors are associated have for some time devoted considerable effort to the study of these transformations, and some of the data are presented here in the expectation that they will be of interest to the tool steel consuming industry. Since tools and dies occasionally crack when they are heat treated, and since it is reasonable to expect that such cracking is associated with the formation of martensite, it is essential, for the full under-

---

A paper presented before the Twenty-fourth Annual Convention of the Society, held in Cleveland, October 12 to 16, 1942. Of the authors, Peter Payson is associated with the Eastern Research Laboratory, Crucible Steel Company of America, Harrison, N. J., and lecturer in metallurgy on the faculty of the Graduate School of Stevens Institute of Technology, Hoboken, N. J., and J. L. Klein is associated with the Eastern Research Laboratory, Crucible Steel Company of America, Harrison, N. J. Manuscript received July 15, 1942.

standing of these tool steels, that particular attention be paid to the low temperature portions of the transformation curves.

The method used to establish the upper parts of the transformation curves is by this time fairly familiar (1), (2), (3).[1] It consists mainly of heating small samples of the steel to austenitizing temperatures; quickly transferring the austenitized samples to baths at various temperatures below the critical temperature of the steel; holding them at these temperatures for various times; quenching them in water; and finally examining them for hardness and microstructure. The times for the beginning and end of transformation at any particular subcritical temperature are then plotted on semilogarithmic paper, and curves are drawn through the points to represent the beginning and end of transformation over a range of temperatures.

Experimental Procedure

Since the information sought in this work was to be of practical rather than academic value, no attempt was made to place the steels in more or less ideal, i.e., homogeneous, conditions, before transformation reactions were measured. The steels were annealed commercially, and then austenitized at temperatures which are ordinarily used when these steels are hardened. For the same practical reason, holding at subcritical temperatures was limited to about 15 to 25 hours although many of these steels failed to transform within this time at many of the temperatures studied. Furthermore, in order to make the curves easy to use, the time is given in the practical units, minutes and hours, rather than in the more conventional values, like 50,000 seconds.

The progress of transformation at temperatures above about 500 degrees Fahr. (260 degrees Cent.) can be followed in most cases readily enough by means of the microstructures of samples quenched after increasing holding times at any one temperature, because the transformation product etches quite differently from the martensite formed from the untransformed austenite when the sample is quenched. However, at lower temperatures the transformation product usually cannot be distinguished from the martensite. Furthermore, the hardnesses of low temperature transformation products are practically the same as that of martensite, and therefore the progress

[1]The figures appearing in parentheses refer to the bibliography appended to this paper.

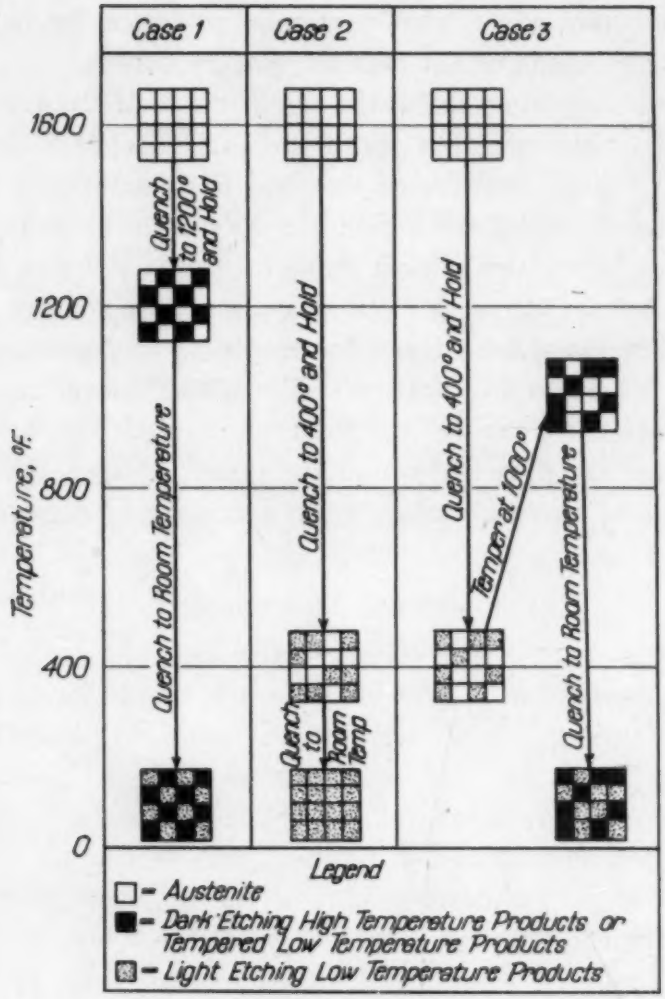


Fig. 1—Reactions Which May Occur During Transformation of Austenite. Cases 1 and 2 are Conventional Procedures. Case 3 is Referred to as "Quench-Temper" Procedure.

of transformation at these temperatures cannot be followed by means of hardness tests.

A method suggested by Greninger and Troiano (4), (5) proved highly successful for studying the low temperature reactions in all of the tool steels from the plain carbon to the highly alloyed high speed steels. Small samples are austenitized; quenched to, and held at, the temperature at which transformation reactions are to be measured, then, instead of being quenched to room temperature, they are transferred to a bath at a higher temperature, appropriate for tempering the transformation product of the particular steel; and, finally, are quenched in water at room temperature. The reactions which take place in this hot quench-temper procedure are perhaps

best explained by means of the diagram, Fig. 1. The first column gives schematically the behavior during the transformation of austenite at high temperatures—the transformation product is such that it can readily be distinguished from the martensite when the partially transformed piece is quenched to room temperature and then examined for structure. The second column shows the transformation at a low temperature—the transformation product blending completely with the martensite when the partially transformed piece is quenched to room temperature and then examined for structure. The third column, in its first step, is similar to that of the second column. However, the additional step of tempering causes the transformation product to etch dark so that when the partially transformed piece is quenched to room temperature, and examined for structure, the transformation product can be distinguished from the martensite (and retained austenite) in nearly all cases. In a few instances, even this procedure is not altogether satisfactory, and it becomes necessary to rely on hardness, rather than on microstructure, to distinguish the sample which has transformed from that which has not, the lower the hardness after the tempering treatment, the greater the amount of transformation. Obviously, in this quench-temper procedure, the selected tempering temperature must be first, a temperature at which the austenite does not transform in a short time, and second, a temperature at which, in the given short time, the transformation product is affected sufficiently so that it etches dark. Examples of suitable tempering temperatures will be given in the following discussions.

In the paper by Greninger and Troiano referred to previously, and in subsequent discussions (6), (7), it was emphasized that the formation of martensite from austenite was not an isothermal reaction, but a reaction which depended for its completion on a decrease in temperature; that martensite began to form immediately as soon as the austenite cooled to a certain temperature, and that the reaction continued only as long as the austenite continued to cool. Since, in the work reported here, it was of interest to check the progress of the martensite formation, considerable attention was paid to the time required to cool the samples to the temperatures of the different quenching baths. In the first place, in order to minimize the time required to cool the specimen to the low transformation temperatures, the samples used were of relatively small dimensions, namely, 0.035 to 0.250 inch in thickness, and in addition, they were precooled

by a quench into a bath at a temperature not far above the transformation temperature. For example, if the piece was to be transformed at 350 degrees Fahr. (175 degrees Cent.), it was first quenched into a bath at about 600 degrees Fahr. (315 degrees Cent.), and held there for 5 to 10 seconds, and then quenched into the bath at 350 degrees Fahr. (175 degrees Cent.). In the second place, the times required for samples of different sizes to cool from the precooling temperature to different temperatures below 400 degrees Fahr. (205 degrees Cent.) were measured, so that in the consideration of time at these different transformation temperatures, a correction could be made for the time required for the specimen to cool to the bath temperature. These cooling times were measured by thermocouples welded to specimens similar in size to those used for the transformation studies. These data, given in Table I, show that even the small specimens used do not instantaneously attain the temperatures of the baths into which they are quenched, but that a definite time must be allowed for the specimen to get to the bath temperature.

Table I

Cooling Times of Three TTT Specimens

Time to get within 5 degrees Fahr. of bath temperature measured with Chromel-Alumel thermocouple welded to outside of specimen

Specimen 0.035 x ⅜ x ⅜"	From 550°F. to	350°F.	300°F.	180°F.	180°F.	80°F.	80°F.	80°F.
	In bath of	*W.M.	W.M.	W.M.	Oil	Oil	Water	Brine
	Time in seconds	3	3	3½	8	10	2½	2½
Specimen ⅛ x ⅜ x ½"	From 600°F. to	500°F.	450°F.	400°F.	350°F.	300°F.	200°F.	80°F.
	In bath of	Salt	Salt	W.M.	W.M.	W.M.	Oil	Water
	Time in seconds	6	7	6	6	6	16	4
Specimen ¼ x ¼ x ½"	From 600°F. to	400°F.	350°F.	300°F.	200°F.	80°F.		
	In bath of	W.M.	W.M.	W.M.	Oil	Water		
	Time in seconds	9	9	9	18	5		

*W.M.—Wood's Metal.

An additional precaution throughout these determinations was the use of control samples to establish whether the particular precooling, and tempering, temperatures could be permitted without giving misleading results due to any possible transformation of austenite during the precool, or tempering, treatments.

The austenitizing treatments, and the tempering treatments in the quench-temper procedure, used for the different steels to establish their transformation temperature time curves are given in Table II, together with the analyses of the steels.

Table II
Austenitizing and Tempering Treatments Used in Determination of TTT Curves

Grade of Steel	Per Cent C	Mn	Si	Cr	V	Mo	W	Co	Austenitized at °F.	for Time	In Quench-Temper Procedure, Tempered at °F.	for Time
Extra Tool	1.14	0.22	0.16	....	....	....	....	....	1450	1 hr.	550	30 sec.
C-Cr Tool	1.01	0.50	0.30	1.21	....	....	....	....	1500	30 min.	650	10 sec.
W. Fast Finishing	1.32	0.28	0.50	0.22	....	....	3.51	....	1550	1 hr.	600	45 sec.
Oil Hardening Non-deforming	0.85	1.18	0.26	0.50	....	....	0.44	....	1450	1 hr.	600	30 sec.
Cr-Mo-W Hot Work	0.32	0.35	0.95	4.86	....	1.45	1.29	....	1850	1 hr.	1050	1 hr.
5 Cr Air Hardening	0.97	0.48	0.40	4.58	0.25	1.04	....	....	1850	1 hr.	1000	3 min.
High C High Cr Air Hardening	1.55	0.27	0.45	11.34	0.24	0.53	....	....	1800	1 hr.	1050	30 min.
18-4-1 High Speed	0.72	0.27	0.39	4.09	1.25	....	18.59	....	2350	2 min.	1050	1 hr.
18-4-1 plus Co High Speed	0.72	0.23	0.43	4.04	1.24	....	18.38	4.72	2350	2 min.	1050	1 hr.
5-4-4-1 High Speed	0.83	0.32	0.25	3.89	1.30	4.30	5.79	....	2225	2 min.	1050	1 hr.
5-4-4-1 plus Co High Speed	0.81	0.41	0.31	4.11	1.51	4.27	5.46	5.22	2200	2 min.	1050	1 hr.

Discussion of Results

The TTT curves of these steels are given in Figs. 2 to 12. The upper portions of the curves are represented conventionally, the line at the left indicating approximately the time required for the particular austenite to begin to transform at any subcritical temperature, and the line at the right, the approximate time for the austenite to be completely transformed at any temperature. The numbers adjacent to the end-of-transformation line represent the Rockwell C hardnesses of the transformation products formed at the different temperatures.

The manner of drawing the lower portions of the curves is new and requires more explanation. It was found, by means of the quench-temper procedure, that the austenite of each steel begins to transform to martensite as soon as it cools to a specific temperature, and that the continuation of the transformation of austenite to martensite depends on the continued cooling of the austenite. It was found, furthermore, that if the austenite is cooled to a low temperature, and held for a long time at this temperature, some portion of the austenite will transform immediately to martensite, and the balance may eventually transform to bainite.

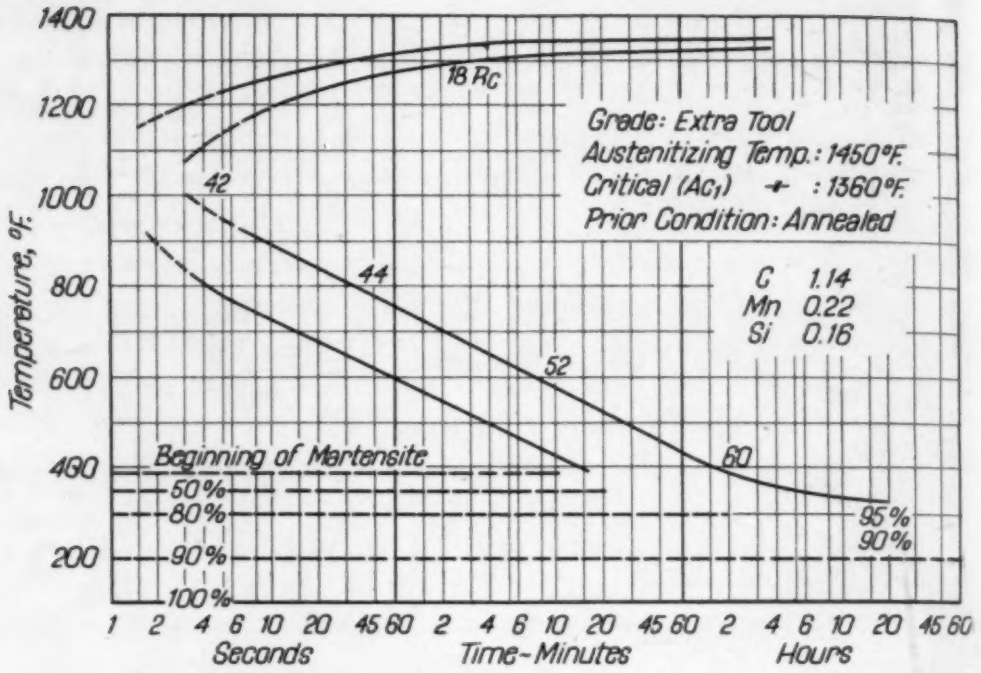


Fig. 2—Transformation Temperature Time Curve of the Austenite of Extra Tool Steel.

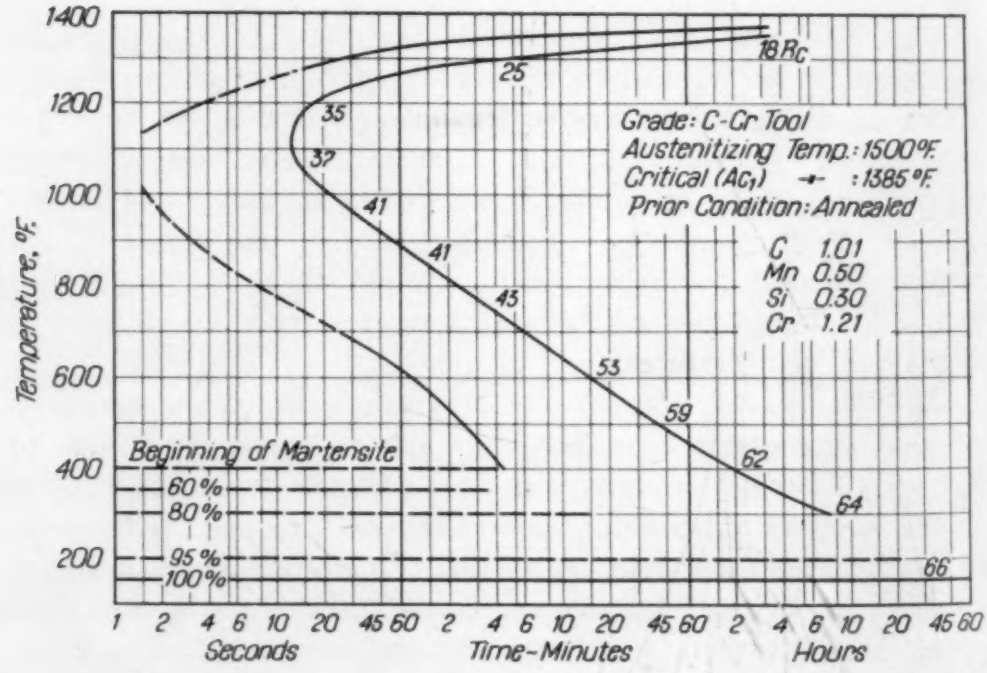


Fig. 3—Transformation Temperature Time Curve of the Austenite of Carbon-Chromium Tool Steel.

The presentation of these reactions in the transformation curves has accordingly been given in a series of horizontal lines, as suggested by Morris Cohen (8). The first horizontal—going down the temperature scale—indicates the Ar″ temperature, i.e., the temperature at which austenite begins to transform to martensite. The subsequent horizontal lines indicate the approximate amount of martensite which has formed when the austenite has been cooled to these temperatures. For example, in Fig. 2, the line at 350 degrees Fahr. (175 degrees Cent.) indicates that extra tool steel, austenitized at 1450 degrees Fahr. (785 degrees Cent.) and cooled rapidly enough to avoid high temperature transformation, will consist of about 50 per cent martensite and 50 per cent austenite (aside from residual carbides) as soon as it has cooled to 350 degrees Fahr. (175 degrees Cent.); the 200 degrees Fahr. (95 degrees Cent.) line indicates that it will consist of about 90 per cent martensite and 10 per cent austenite as soon as it has cooled to 200 degrees Fahr. (95 degrees Cent.); and the 80 degrees Fahr. (25 degrees Cent.), or room temperature line, that it will consist of 100 per cent martensite as soon as it has cooled to 80 degrees Fahr. (25 degrees Cent.). Furthermore, the right ends of the horizontal lines indicate the times at which isothermal transformation will start at these temperatures if the austenite is allowed to remain at these temperatures for long periods. For example, in Fig. 2, the 350 degrees Fahr. (175 degrees Cent.) line ends at 20 minutes. This means that if the austenite of this steel is cooled to 350 degrees Fahr. (175 degrees Cent.) and held at this temperature, about 50 per cent of the austenite will transform immediately to martensite, and the residual 50 per cent will begin to transform isothermally in about 20 minutes, and will continue to transform until about 4 hours elapse, at which time the austenite will be completely transformed, as indicated by the end-of-transformation line. Further, the 300 degrees Fahr. (150 degrees Cent.) line in Fig. 2 ends at 2 hours. At this temperature, then, the austenite which has not transformed to martensite begins to transform isothermally in about 2 hours, and continues to transform as long as it is held at this temperature. However, as indicated on the curve sheet, the rate of transformation at this temperature is quite slow since only about 95 per cent is complete at the end of about 15 hours.

Thus, the lower portions of all the curves show the manner in which martensite forms from austenite as the austenite is cooled

continuously to room temperature, and also the manner in which the austenite may transform if it is cooled to a low temperature and held for a long time at this temperature. The percentages indicated on these curves are not to be taken too literally—they are, frankly, reasonable approximations, made by observers who have had considerable experience in examining microstructures of partially transformed sections. No effort was made to get accurate quantitative data on these mixed structures, because the task is tremendous even for the simple extra tool steel, and it is further complicated in the higher alloy steels by the fact that the alloy-rich portions in the regions of carbide streaks in the steels do not transform at the same rate as portions relatively free of residual carbides. Furthermore, there appears to be no practical need for accurate determinations of the amounts of transformed, and untransformed, austenite. From the practical viewpoint, it is not the exact amount of untransformed austenite which is important, but rather the fact that some austenite remains untransformed at a particular point in the heat treating operation. It is the authors' opinion that retained austenite in amounts as small as 5 per cent is readily detected by the quench-temper procedure described above, and that therefore conclusions may safely be based on the estimated amounts given on the curve sheets.

Two groups of photomicrographs are given in Figs. 13 to 16, and Figs. 17 to 21, to illustrate the formation of martensite in carbon-chromium tool steel, and in 18-4-1 high speed steel, respectively. The progress of martensite formation is similar in all the steels, but the amounts present at any temperature differ to some degree in the various steels, as indicated on the curve sheets. The photographs also serve to show the authors' method of estimating percentages of transformed product. However the values reported for any condition are based on the examination of many fields, rather than just one, because of the streaks in most of these steels.

As a matter of interest, Figs. 22 and 23 are shown to illustrate the formation of both martensite and isothermal product at the same temperature. Although the photomicrographs represent 18-4-1 high speed steel, similar behaviors were found in all the tool steels. The martensite generally has the appearance of feathers, whereas the isothermal product is generally much more needlelike.

Since the subject of this paper is the hardening of tool steels, the reactions which occur at high temperatures will be given only

little consideration. The upper portions of the curves serve to corroborate the well known facts that extra tool steel, carbon-chromium tool steel, and tungsten fast finishing steel, Figs. 2, 3, and 4, must be cooled very rapidly through the temperature range from about 1200 to 800 degrees Fahr. (650 to 425 degrees Cent.), in order to prevent the austenites of these steels from transforming to relatively soft, high temperature products; that the oil hardening, nondeforming steel, Fig. 5, need not be cooled so rapidly since the austenite of this steel transforms much more slowly at high temperatures; and finally, that the higher alloy die steels, and the high speed steels, Figs. 6 to 12, may be hardened even without a rapid quench, since the austenites of these steels react very sluggishly at high temperatures. In other words, the curves explain why the first three steels are quenched in water when they are to be hardened; the fourth, in oil and the other seven in oil, or in air. However, of more importance in this discussion are the reactions which occur below the Ar″, or martensite, temperature.

The curves clearly show that the austenites of these steels are not transformed completely, or nearly so, until they have been cooled close to room temperature, and that, in the cases of some of the more highly alloyed steels, as much as 5 to 10 per cent austenite may be retained untransformed even at room temperature. This is the most significant disclosure of these tool steel studies, and its importance cannot be over-emphasized. It is believed that an understanding of these facts should help to eliminate many of the tool steel failures which occur all too frequently. As long ago as 1928, DeLong and Palmer (9) stated, "Obviously, no steel can be tempered until it has first been hardened . . . . . Many unexplained failures in high speed tools can be traced to their removal from the quenching bath at too high a temperature and then attempting to temper them before the steel has fully hardened . . . . . A good average temperature (to which the steel should be cooled before it is tempered) is that at which the tools can be just about juggled in the bare hand." Many a skilled hardener today uses the rule, "Don't temper the steel after the quench until you can shake hands with it." But in spite of these admonitions, there are still many recommended practices (10) for the heat treatment of tools which include statements like, "Do not cool the steel (in the quench) below the temperature of boiling water. Reheat immediately to the tempering temperature"; "The (tungsten high speed) tool is then

cooled slowly in air to 200 to 300 degrees Fahr. (95 to 150 degrees Cent.), then tempered immediately to avoid cracking"; "Dies should be tempered immediately after quenching". A consideration of the TTT curves presented here should make it clear why these steels should be cooled close to room temperature before they are tempered, and why cracking is frequently due to the fact that the quenched steel is put into the tempering furnace, or bath, too soon, rather than not soon enough.

The water hardening steels, when treated in small sections, are not likely to be tempered too soon because they would be cooled to low temperatures quite rapidly, in the water quench, and they would be practically completely transformed as soon as they cooled to about 150 degrees Fahr. (65 degrees Cent.). However, when these steels are treated in large sections, a serious condition may arise which can —and frequently does—cause the pieces to crack. Consider, for example, a piece about 6 by 6 by 3 inches, which is hardened on the 6 by 3-inch face, by means of a high pressure spray. The steel immediately in contact with the spray will be cooled rapidly and will be transformed completely to martensite. The steel at the back of the block will cool very much more slowly, and will transform completely to very fine pearite at temperatures between about 1100 and 900 degrees Fahr. (595 and 480 degrees Cent.) since the transformation at these temperatures is quite rapid. Somewhere between the hardened face and the back will be a zone which must be at about 250 to 400 degrees Fahr. (120 to 205 degrees Cent.) for some time after the spray is applied.

According to the TTT curves, the austenites of these steels do not transform rapidly in this temperature range, i.e., 250 to 400 degrees Fahr. (120 to 205 degrees Cent.), and therefore the steel in that part of the block will remain austenite for some time. Suppose that the block is transferred now to a tempering furnace at 350 or 375 degrees Fahr. (175 or 190 degrees Cent.), before the portion back of the hardened face has had a chance to cool to much below 300 degrees Fahr. (150 degrees Cent.). Since the austenite which had not previously transformed would not transform completely at 350 to 375 degrees Fahr. (175 to 190 degrees Cent.) except after a very long time, according to the TTT curve, there would still be austenite in the block when it is removed from the tempering furnace after the usual tempering time of 1 to 2 hours at temperature. This austenite would finally transform to martensite when the

block cooled to room temperature, and the block would therefore be in a highly stressed condition, in the region where the martensite had formed during the final cooling from the tempering furnace. Although such a condition is not always disastrous, it frequently causes corners of the block to chip off, and sometimes this chipping occurs many hours after the block has cooled to room temperature. To avoid this condition, it is obvious that the steel throughout the block should be allowed to transform completely before it is placed in the tempering furnace. However, if the block is allowed to go cold completely in the quench, it may crack, probably because the cold, hard metal at the outside of the block does not yield sufficiently to the stresses set up when the last portion of the austenite within the block transforms to martensite, with an accompanying increase in volume. A double temper is usually a safe practice in this dilemma, providing the steel is allowed to cool to room temperature after the first temper. A compromise treatment which is known to be successful in the hardening of large dies of water hardening steel is one recommended in the A.S.M. Metals Handbook as follows: "Usually tools should not be removed from the quenching bath until they are below 200 degrees Fahr. (95 degrees Cent.). . . . . neither should they be allowed to go cold in the quench. A good time to remove the tools is when all of the surface is approximately 125 to 150 degrees Fahr. (50 to 65 degrees Cent.). Then, if any mass is involved, hold at this temperature until the tools become uniform in temperature throughout. This may be accomplished by placing in an oil bath for the required length of time".

In the hardening of the oil hardening and air hardening steels, the transformations which occur through the section of the steel are much more uniform than they are in the water hardening steels. This is true because the temperature differences between the outside and the center of the section are much smaller in the oil quench, and air cool, than they are in the water quench. Furthermore, the transformations at high temperatures are so sluggish in these steels that practically all of the austenite transforms at temperatures below the martensite point of the steel, usually below 400 degrees Fahr. (205 degrees Cent.). The cooling rates from 400 degrees Fahr. (205 degrees Cent.) to room temperature are quite slow in warm quenching oil, and in air, and large pieces may require considerable time before they are cooled to temperatures below 200 degrees Fahr. (95 degrees Cent.). As shown in the TTT curves, Figs. 5, 6, and

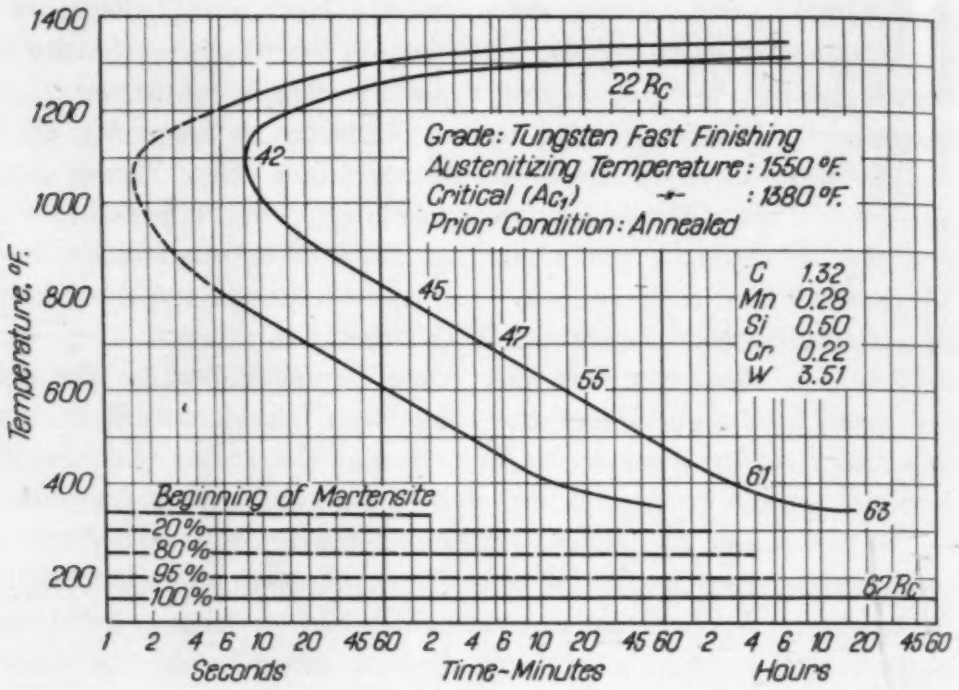


Fig. 4—Transformation Temperature Time Curve of the Austenite of Tungsten Fast Finishing Steel.

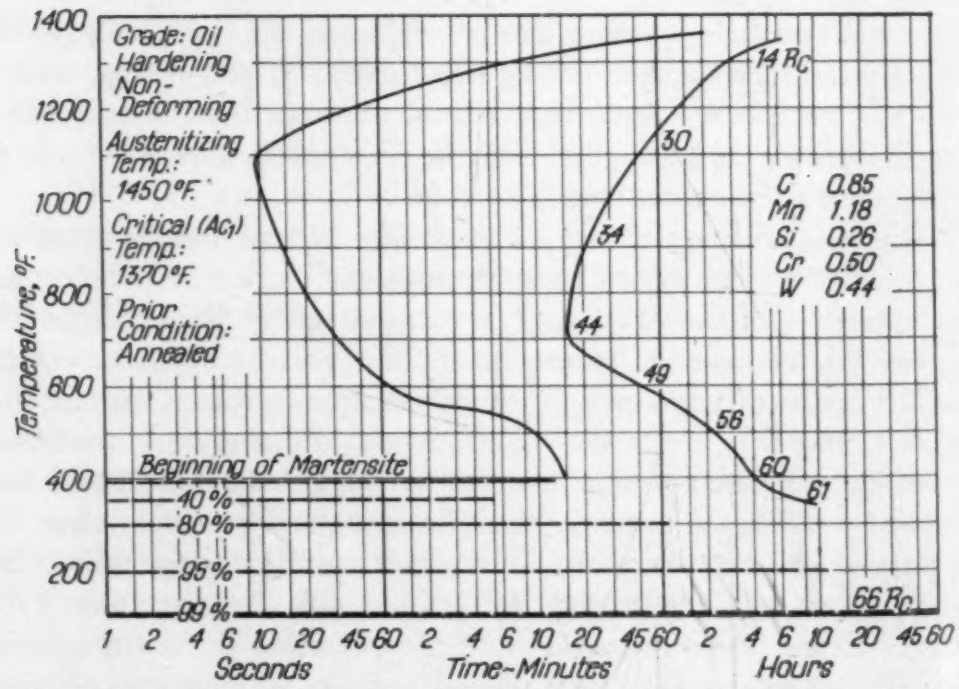


Fig. 5—Transformation Temperature Time Curve of the Austenite of Oil Hardening Nondeforming Steel.

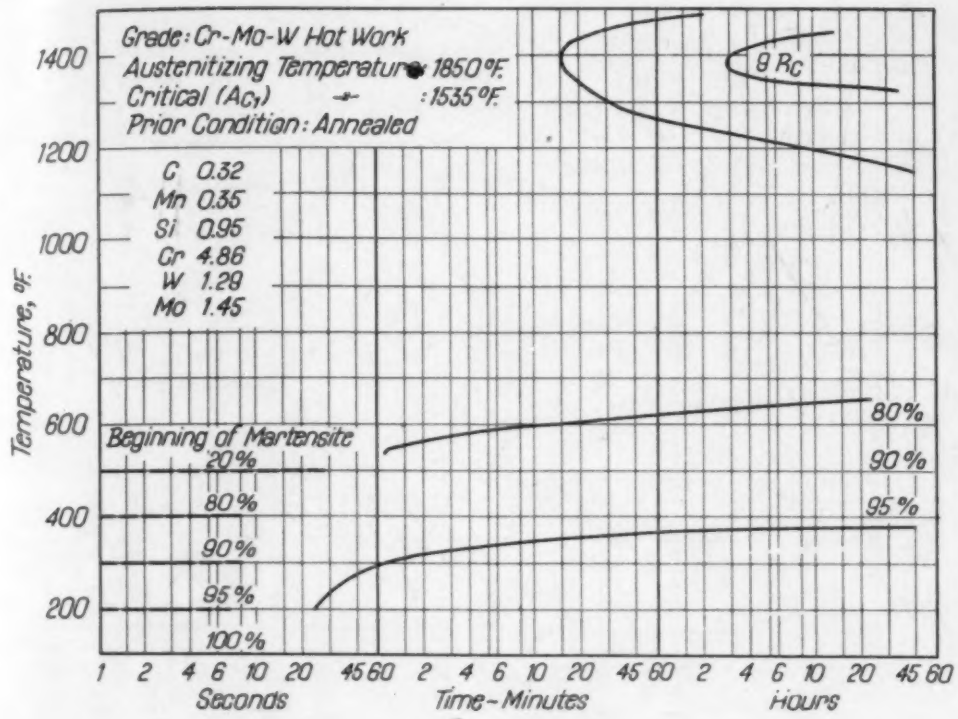


Fig. 6—Transformation Temperature Time Curve of the Austenite of Chromium-Molybdenum-Tungsten Hot Work Steel.

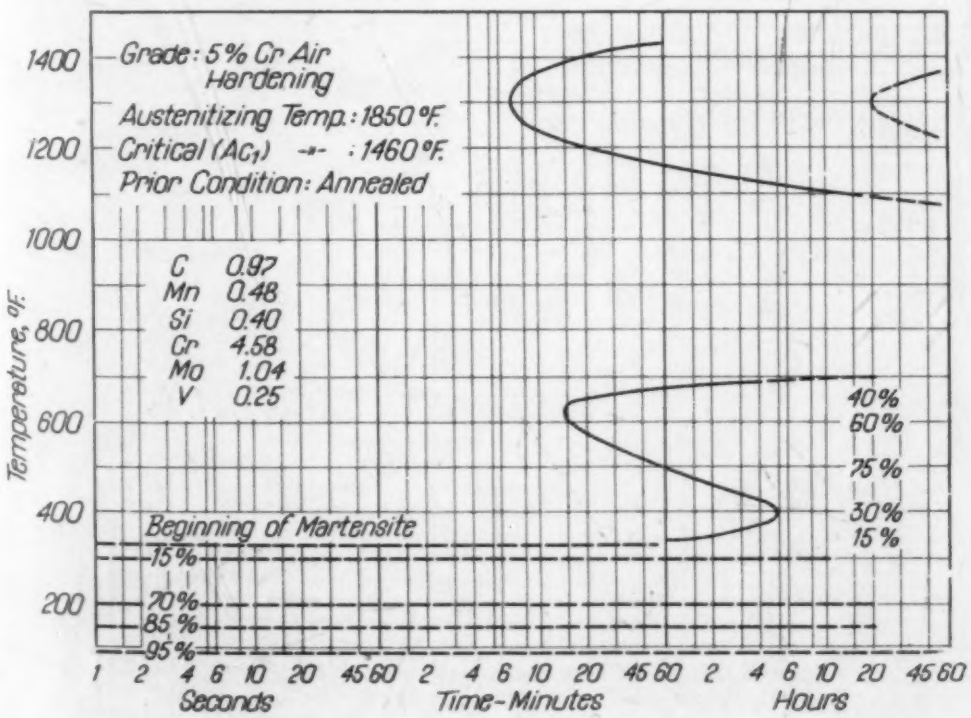


Fig. 7—Transformation Temperature Time Curve of the Austenite of 5 Per Cent Chromium Air Hardening Steel.

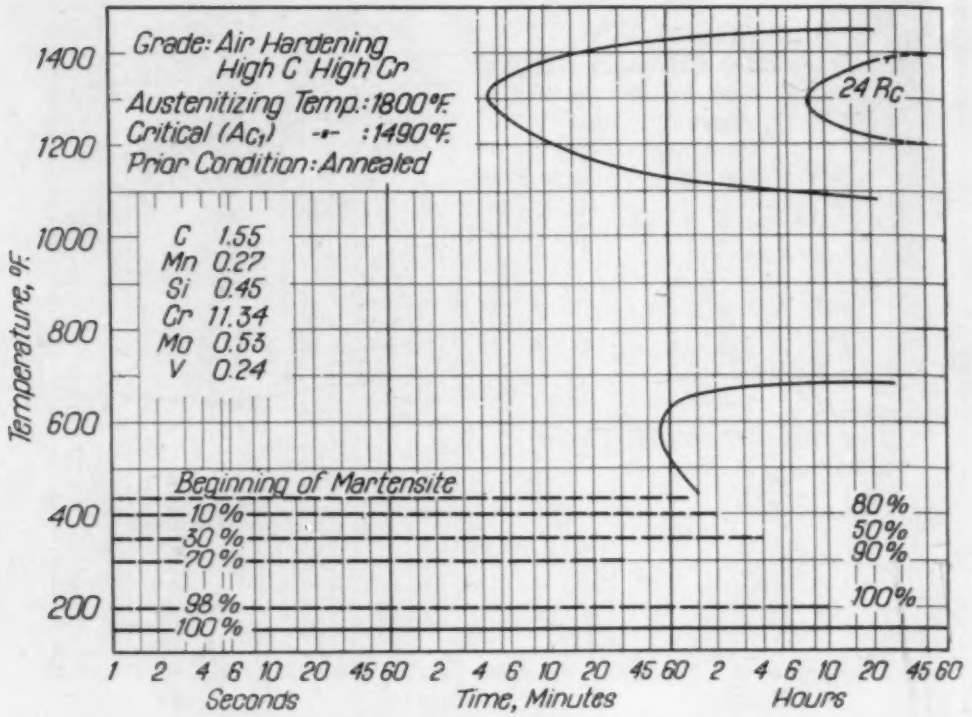


Fig. 8—Transformation Temperature Time Curve of the Austenite of Air Hardening High Carbon-High Chromium Steel.

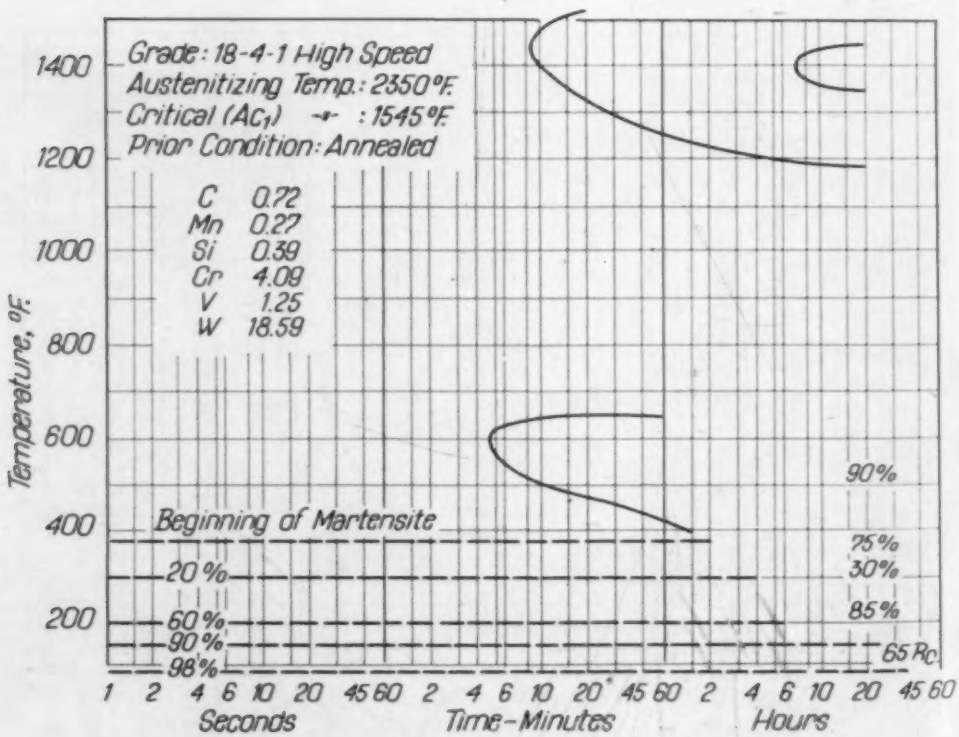


Fig. 9—Transformation Temperature Time Curve of the Austenite of 18-4-1 High Speed Steel.

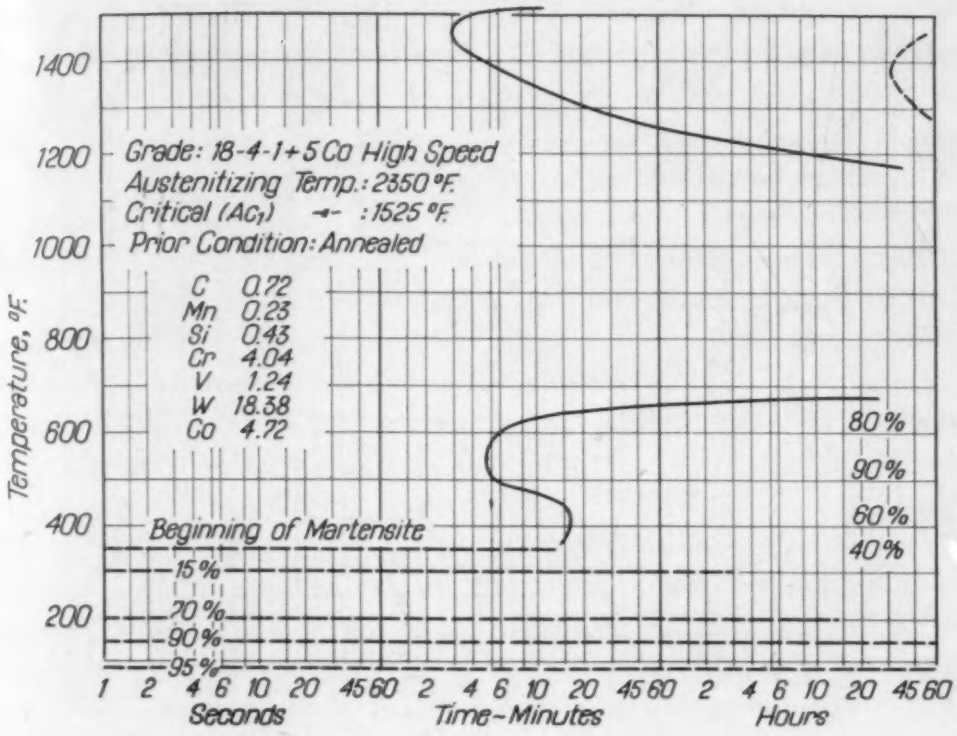


Fig. 10—Transformation Temperature Time Curve of the Austenite of 18-4-1 + Cobalt High Speed Steel.

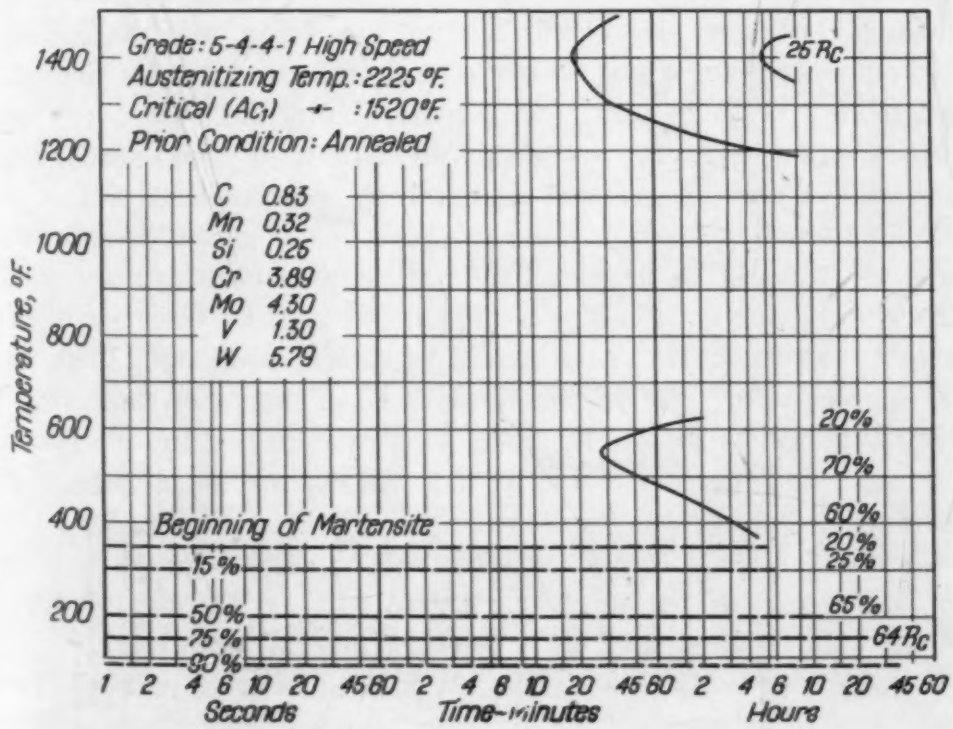


Fig. 11—Transformation Temperature Time Curve of the Austenite of 5-4-4-1 High Speed Steel.

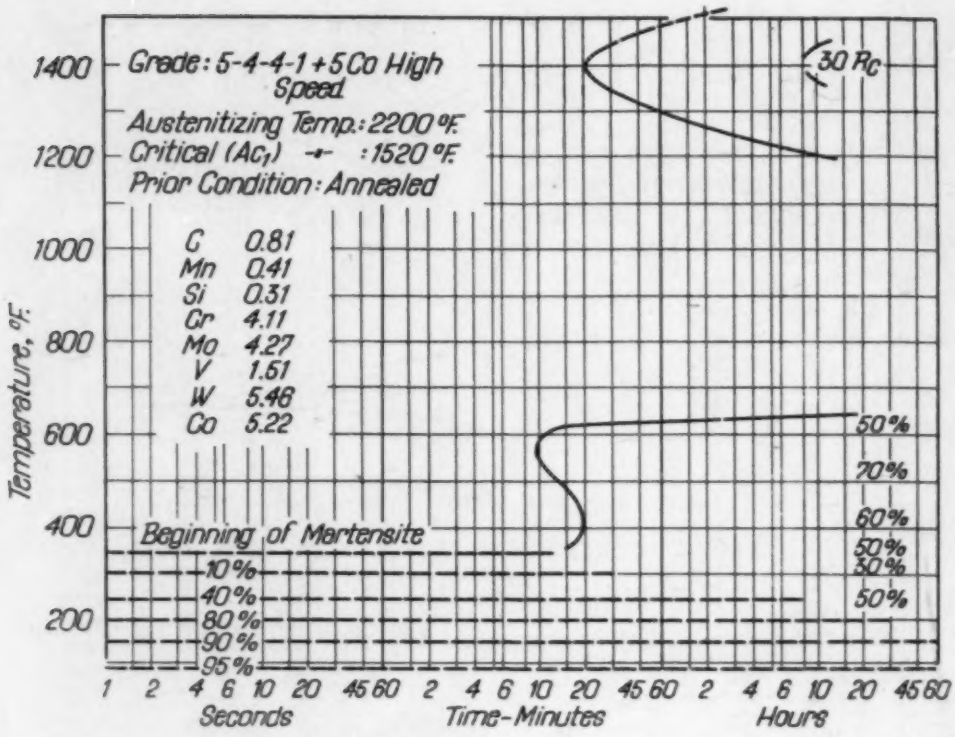


Fig. 12—Transformation Temperature Time Curve of the Austenite of 5-4-4-1 + Cobalt High Speed Steel.

8, the oil hardening nondeforming steel, the chromium-molybdenum-tungsten, hot work steel, and the air hardening high carbon-high chromium steel are about 95 per cent transformed by the time they cool to 200 degrees Fahr. (95 degrees Cent.), but the 5 per cent chromium air hardening steel, and the four high speed steels, Figs. 7, 9, 10, 11 and 12, are still 20 per cent or more austenite at 200 degrees Fahr. (95 degrees Cent.). Tools made of the first three of these steels could be tempered satisfactorily as soon as they cooled throughout to about 150 degrees Fahr. (65 degrees Cent.) but tools made of the other five steels should be cooled throughout to at least 100 degrees Fahr. (40 degrees Cent.) before they are tempered. If they are tempered before the austenite is practically completely transformed, the supposedly tempered tool will contain some untempered martensite when it cools to room temperature after the tempering operation, and (if it does not crack) it will be more brittle than a tool consisting only of tempered martensite (12). The brittleness, of course, may be eliminated by a second tempering treatment.

Some of these steels contain a certain amount of austenite even after they have been cooled to room temperature. Such retained austenite may be "conditioned" by the heating at the tempering tem-

perature, to transform during the cooling to room temperature subsequent to the tempering treatment (13). This is the case in high speed steels because heating at about 1050 degrees Fahr. (565 degrees Cent.) causes precipitation of alloy carbides from the retained austenite, and the austenite is thus changed to a composition which transforms more readily at low temperatures. However, only the hot work, and high speed, steels are ordinarily tempered at temperatures high enough to precipitate alloy carbides and therefore retained austenite in other steels cannot be "conditioned" to transform during the cooling subsequent to the tempering treatment. In some steels, retained austenite may be transformed by tempering if the steels are reheated for a sufficient time at temperatures at which the austenite of the steel will transform isothermally as shown in the TTT curve. For example, according to Fig. 5, any retained austenite in the quenched oil hardening nondeforming steel should be transformed if the steel is reheated to about 375 degrees Fahr. and held at this temperature for about 4 to 6 hours. However, this has not as yet been established experimentally. Some recent publications (14), (15), indicate that the austenite of alloy tool steels tends to become stabilized at low temperatures, and data obtained at the authors' laboratory tend to confirm this. A future paper may deal with the subject of stabilized austenite in these tool steels.

Supplementary Data on the Effect of Austenitizing Temperatures

It has been known for some time, that steels of the high carbon-high chromium type may be made relatively soft and practically nonmagnetic, i.e., almost completely austenitic, at room temperature, when they are heated at about 2000 degrees Fahr. (1095 degrees Cent.) and quenched, whereas the same steels quenched from about 1800 degrees Fahr. (980 degrees Cent.) are quite hard and strongly magnetic, i.e., martensitic, at room temperature. These facts indicated that the austenitizing temperature may have a very marked effect on the manner in which the austenite of tool steels transforms during the quench. Since the quench-temper procedure makes it possible to follow the progress of the formation of martensite at low temperatures, and since tool steels may occasionally be overheated during heat treatment, the authors considered it of interest to study the effect of austenitizing temperatures on the formation of marten-

Table III

Effect of Austenitizing Treatment on the Formation of Martensite in Extra Tool Steel

Austenitizing Treatment	1450° F. 1 Hour	1525° F. 30 Min.	1600° F. 30 Min.
Grain size as established by fracture of hardened sample	9	8½	6
Amount of undissolved carbides	Very many	Many	Some
Per cent Martensite formed at 350° F.	50	5	0
Per cent Martensite formed at 300° F.	80	40	5
Per cent Martensite formed at 200° F.	90	80	50
Per cent Martensite formed at 80° F.	100	95	85

Table IV

Effect of Austenitizing Treatment on the Formation of Martensite in Oil Hardening Nondeforming Steel

Austenitizing Treatment	1450° F. 1 Hour	1525° F. 1 Hour	1600° F. 45 Min.	1800° F. 45 Min.	2000° F. 30 Min.
Fracture Grain Size Number	9¼	9	6½	5	2
Undissolved Carbides	Very many	Many	Few	None	None
Per cent Martensite formed at 350° F.	40	15	0	0	5
Per cent Martensite formed at 300° F.	80	40	10	10	10
Per cent Martensite formed at 200° F.	95	80	40	40	40
Per cent Martensite formed at 80° F.	99	95	90	80	80

Table V

Effect of Austenitizing Temperature on the Formation of Martensite in 5 Per Cent Chromium Air Hardening Steel

Austenitized ½ Hour at	1750° F.	1800° F.	1850° F.	1900° F.
Per cent Martensite formed at 200° F.	95	80	70	50
Per cent Martensite formed at 150° F.	100	85	80	70
Per cent Martensite formed at 80° F.	100	Over 95	95	90

Table VI

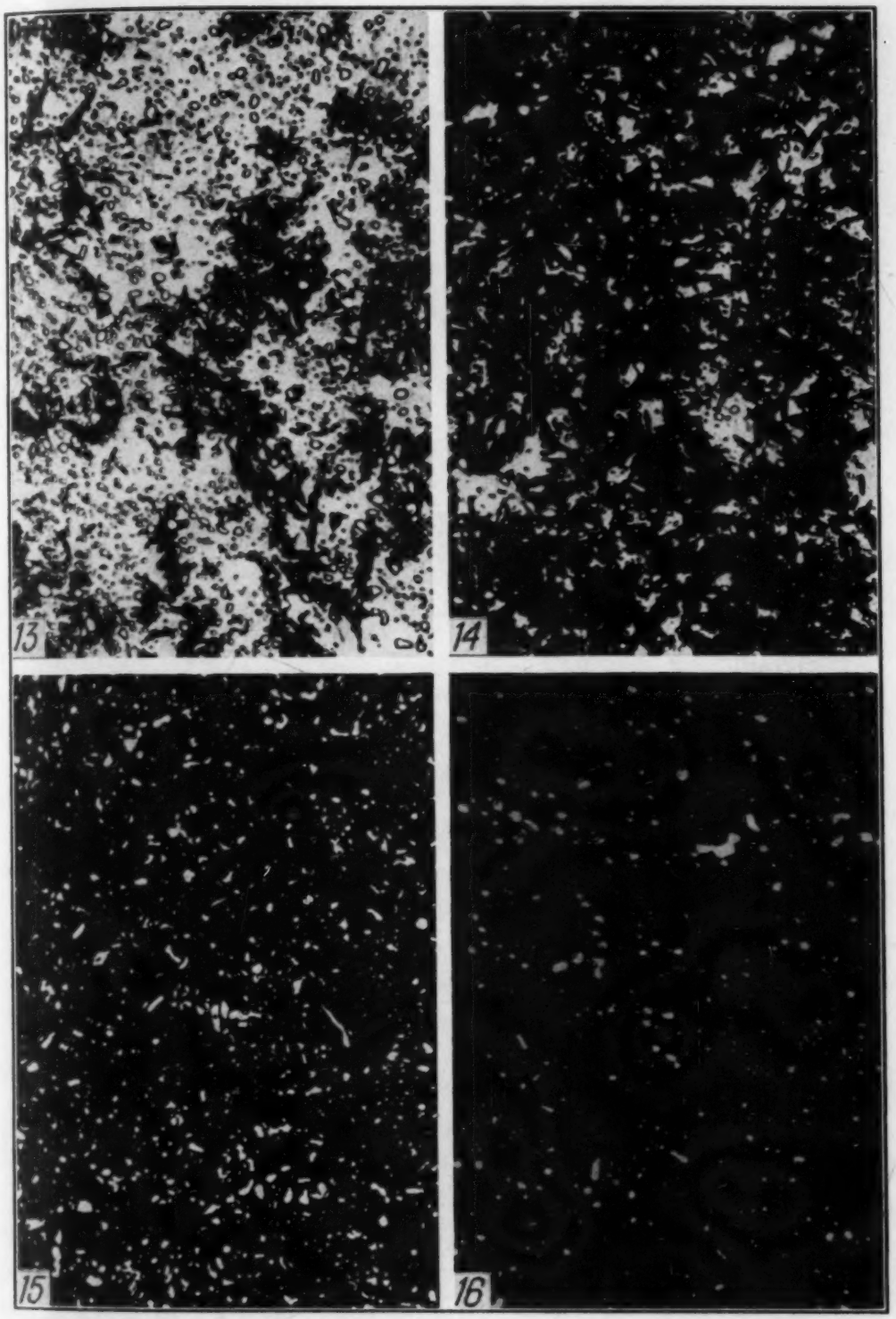
Effect of Austenitizing Treatment on Formation of Martensite in Chromium-Molybdenum-Tungsten Hot Work Steel

Austenitizing Treatment	1800° F. 1 Hour	1900° F. 45 Min.	2000° F. 20 Min.	2100° F. 5 Min.
Grain size of austenite established by carbide network formed at 1450° F.	9	8	6	4
Per cent Martensite formed at 200° F.	95	95	95	95
Per cent Martensite formed at 80° F.	100	100	100	100

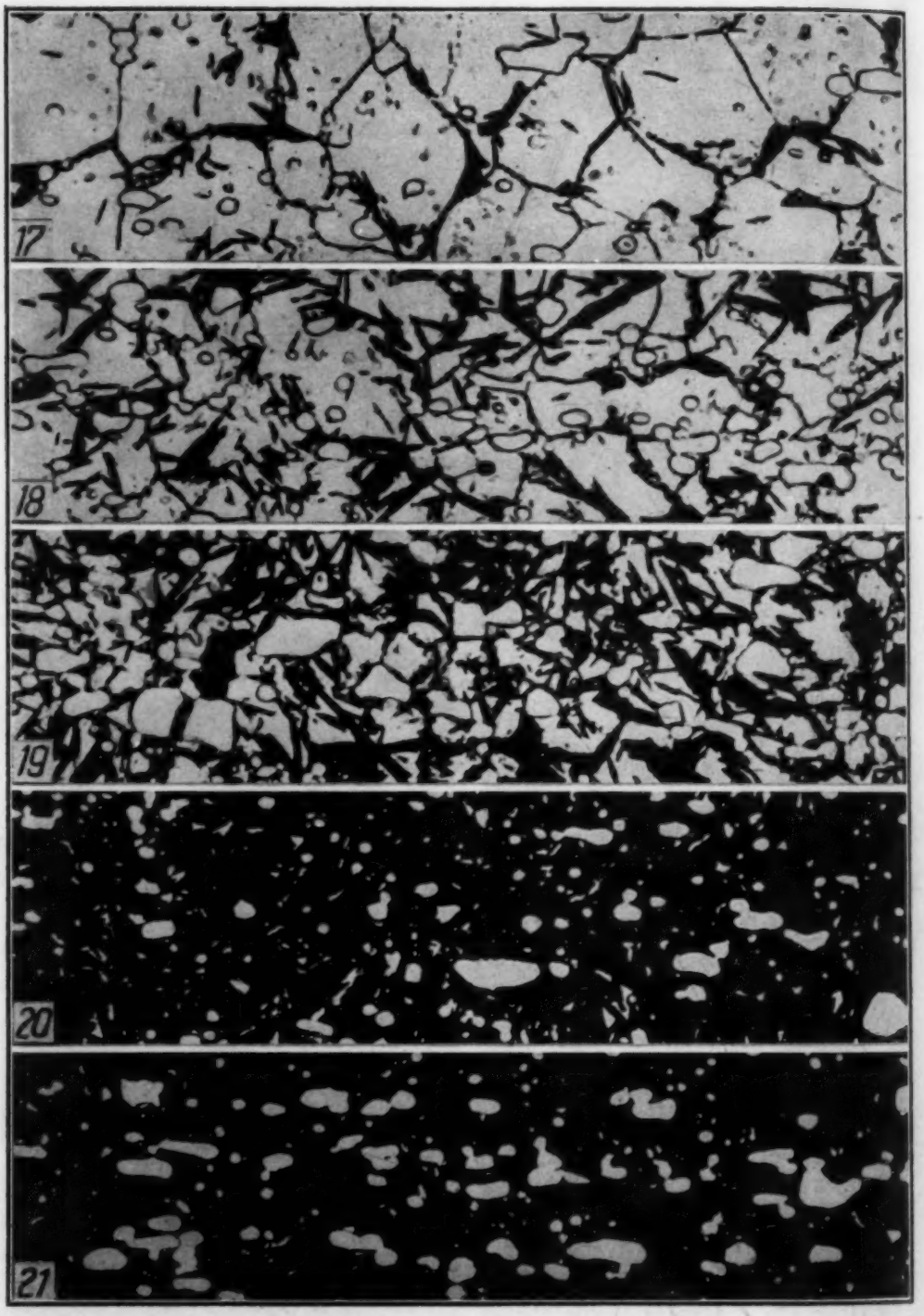
Table VII

Effect of Austenitizing Treatment on Formation of Martensite in Air Hardening High Carbon-High Chromium Steel

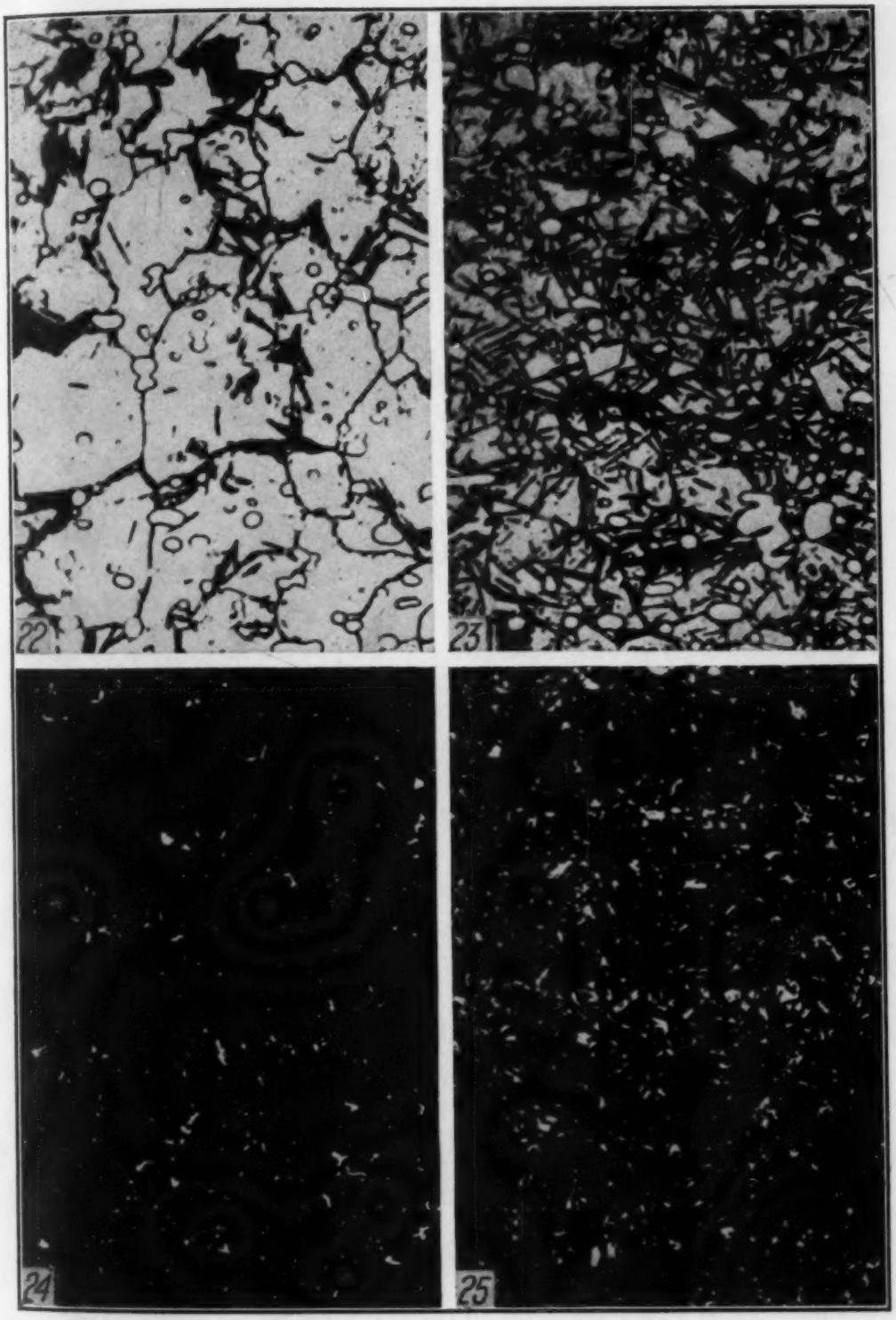
Austenitizing Treatment	1850° F. 1 Hour	1900° F. 45 Min.	1950° F. 30 Min.	2000° F. 20 Min.	2050° F. 10 Min.	2100° F. 5 Min.
Grain size of austenite established by carbide network formed at 1450° F.	..	10½	..	..	..	9
Per cent Martensite formed at 200° F.	95	65	20	5	Trace	0
Per cent Martensite formed at 80° F.	100	Over 95	85	60	5	Trace
Rockwell Hardness as Quenched	C-65	C-64	C-59	C-52	C-41	C-36



Figs. 13-16—Formation of Martensite in Carbon-Chromium Tool Steels. Austenitized at 1500 Degrees Fahr. Etched in 5 Per Cent Nital. × 1000. Fig. 13—Cooled to 380 Degrees Fahr. 20 Per Cent Martensite. Fig. 14—Cooled to 300 Degrees Fahr. 80 Per Cent Martensite. Fig. 15—Cooled to 200 Degrees Fahr. 95 Per Cent Martensite. Fig. 16—Cooled to 150 Degrees Fahr. 100 Per Cent Martensite.

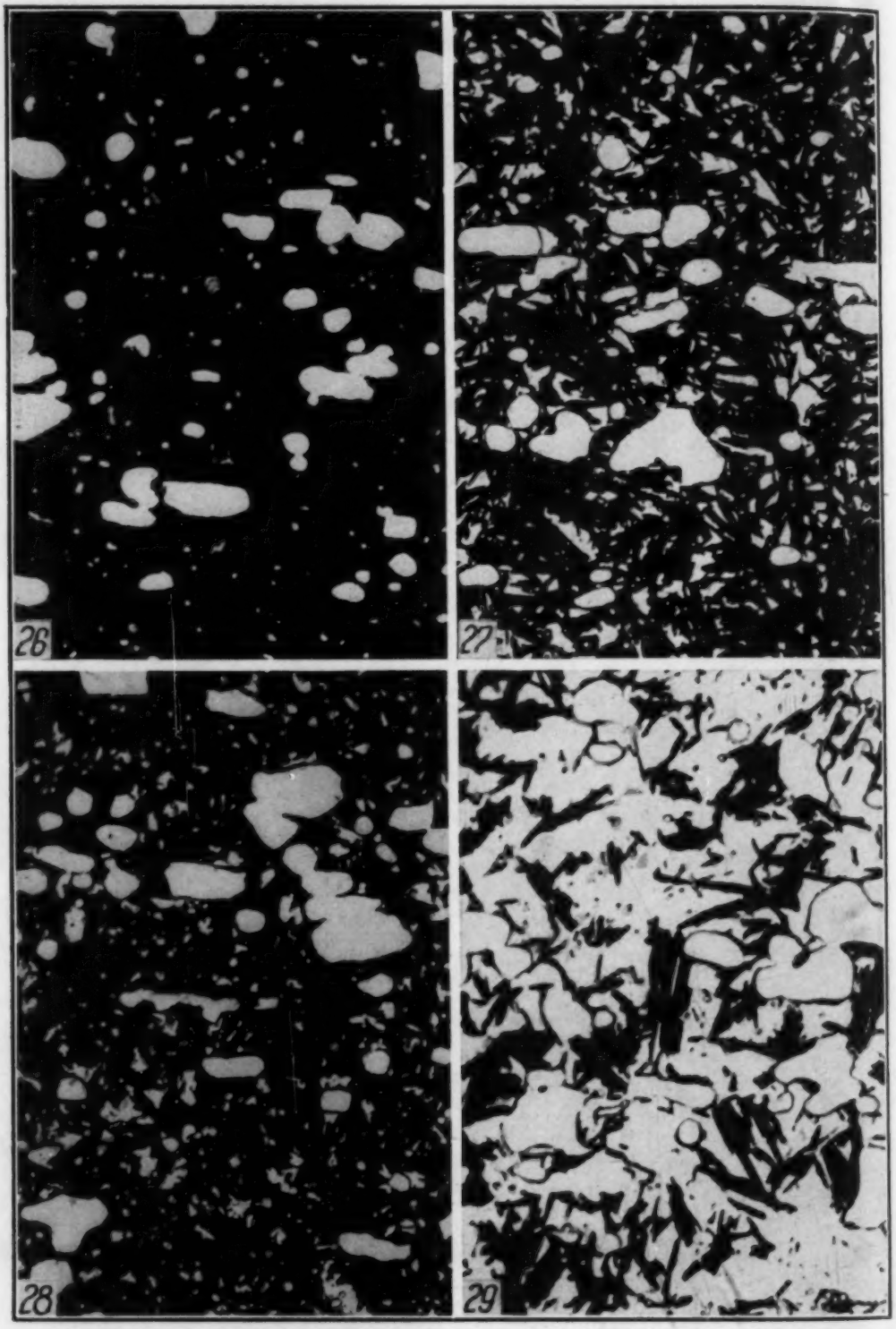


Figs. 17-21—Formation of Martensite in 18-4-1 High Speed Steel, Austenitized at 2350 Degrees Fahr. Etched in 5 Per Cent Nital. × 1000. Fig. 17—Cooled to 350 Degrees Fahr. 5 Per Cent Martensite. Fig. 18—Cooled to 300 Degrees Fahr. 20 Per Cent Martensite. Fig. 19—Cooled to 200 Degrees Fahr. 60 Per Cent Martensite. Fig. 20—Cooled to 150 Degrees Fahr. 90 Per Cent Martensite. Fig. 21—Cooled to 80 Degrees Fahr. 98 Per Cent Martensite.



Figs. 22 and 23—Martensite, and Isothermal Product, Formation at 350 Degrees Fahr. in 18-4-1 High Speed Steel Austenitized at 2350 Degrees Fahr. Etched in 5 Per Cent Nital. × 1000. Fig. 22—Cooled to 350 Degrees Fahr. and Held Only 15 Seconds. 5 Per Cent Martensite. Fig. 23—Cooled to 350 Degrees Fahr. and Held 5 Hours. 5 Per Cent Martensite Plus Isothermal Product.

Figs. 24 and 25—Effect of Austenitizing Temperature on Transformation at Room Temperature of Oil Hardening, Nondeforming Steel. Etched in 5 Per Cent Nital. × 1000. Fig. 24—Austenitized at 1450 Degrees Fahr. Quenched to 80 Degrees Fahr. 99 Per Cent Martensite. Fig. 25—Austenitized at 1600 Degrees Fahr. Quenched to 80 Degrees Fahr. 90 Per Cent Martensite.



Figs. 26-29—Effect of Austenitizing Temperatures on Transformation at 200 Degrees Fahr. and at 80 Degrees Fahr. of High Carbon-High Chromium Air Hardening Steel. Etched in 10 Per Cent Nital. × 1000. Fig. 26—Austenitized at 1850 Degrees Fahr., Quenched to 80 Degrees Fahr. 100 Per Cent Martensite. Fig. 27—Austenitized at 1950 Degrees Fahr., Quenched to 80 Degrees Fahr. 85 Per Cent Martensite. Fig. 28—Austenitized at 1850 Degrees Fahr., Quenched to 200 Degrees Fahr. 95 Per Cent Martensite. Fig. 29—Austenitized at 1950 Degrees Fahr., Quenched to 200 Degrees Fahr. 20 Per Cent Martensite.

site in some of these steels. The steels for this suppplementary investigation were the extra tool steel, the oil hardening nondeforming steel, the 5 per cent chromium air hardening steel, the chromium-molybdenum-tungsten hot work steel, and the air hardening high carbon-high chromium steel. Samples of each steel were austenitized over a range of temperatures, then quenched to temperatures below the martensite points, tempered immediately at appropriate temperatures, and examined microstructurally for percentages of transformed austenite. The data are given in Tables III to VII and some photomicrographs in Figs. 24 to 29.

These data show that the austenitizing treatment may have a pronounced effect on the manner in which martensite forms in these steels, or it may have no effect. The grain size of the austenite apparently has no effect on the martensite formation. This is quite interesting since austenite grain size is known to have a marked effect on hardenability (16), (17). Evidently, the austenite grain size affects the rate of transformation of austenite at temperatures between about 1300 and 900 degrees Fahr. (705 and 480 degrees Cent.), but does not affect the transformation of austenite to martensite. On the other hand, changes in composition of the austenite have very important effects on the formation of martensite. The chromium-molybdenum-tungsten hot work steel is unaffected in its martensite formation by variations in austenitizing temperature from 1800 to 2100 degrees Fahr. (980 to 1150 degrees Cent.) even though these treatments changed the austenite grain size from 9 to 4. The carbides in this low carbon steel are practically completely dissolved at 1800 degrees Fahr. (980 degrees Cent.), and therefore higher temperatures could not affect the composition of the austenite through solution of carbides. All the other steels considered here, on the other hand, have many carbides out of solution at the austenitizing temperatures ordinarily used for hardening them. When these higher carbon steels are heated at temperatures higher than usual, their austenites become increasingly rich in carbon (and in alloying elements like manganese, chromium, tungsten, molybdenum, etc., if they are present) as the austenitizing temperature is raised. Thus, in these steels, the austenite composition changes with increasing temperature until all the carbides are dissolved.

The behavior of the oil hardening nondeforming steel as shown in Table IV is a good illustration of the effects of austenite composition, and austenite grain size, on the formation of martensite. When

this steel is austenitized at increasing temperatures from 1450 to 1600 degrees Fahr. (790 to 870 degrees Cent.), there is a gradual increase in austenite grain size, and a continuous change in austenite composition due to solution of carbides, and these changes are accompanied by a retardation of the formation of martensite at 200 degrees Fahr. (95 degrees Cent.) and at 80 degrees Fahr. (25 degrees Cent.). Between 1600 and 2000 degrees Fahr. (870 and 1095 degrees Cent.), the austenite of this steel continues to grow to a very coarse size, but the composition remains about the same since the carbides were nearly completely dissolved at 1600 degrees Fahr. (870 degrees Cent.). This increase in austenitizing temperature from 1600 to 2000 degrees Fahr. (870 to 1095 degrees Cent.) hardly affects the formation of martensite at 200 degrees Fahr. (95 degrees Cent.) and at 80 degrees Fahr. (25 degrees Cent.). The steel now behaves like the low carbon chromium-molybdenum-tungsten hot work steel in respect to the effect of austenitizing temperature on the formation of martensite.

The behavior of the air hardening high carbon-high chromium steel, as shown in Table VII, emphasizes the marked effect of changes in austenite composition on the formation of martensite. This steel shows only a small increase in grain size with temperature increase from 1850 to 2100 degrees Fahr. (1010 to 1150 degrees Cent.) but the effect of increasing austenitizing temperature on the formation of martensite at 200 degrees Fahr. (95 degrees Cent.), and at 80 degrees Fahr. (25 degrees Cent.), is very great. This steel has many excess carbides not only at 1850 degrees Fahr. (1010 degrees Cent.), but also at 2100 degrees Fahr. (1150 degrees Cent.). Although the increases in solution of carbide over this range of austenitizing temperatures are not easy to recognize in the microstructures, it is reasonable to infer that the austenite of this steel does dissolve increasing amounts of carbide at increasing temperatures. With the increase in carbon dissolved in the austenite, there must also be an increase in the amount of chromium, and perhaps molybdenum, dissolved in the austenite. The higher carbon and higher alloy content of the austenite causes the martensite formation to be depressed to lower and lower temperatures—or, conversely, the higher carbon and higher alloy content of the austenite causes the amount of austenite retained at room temperature to be greater and greater.

There are far too few data as yet from which to draw conclusions on the effects of various elements dissolved in austenite, on the

transformation of the austenite at low temperatures. The authors venture the opinion, on the basis of the data presented here, as well as on other data, that carbon is one of the most effective elements in retarding the formation of martensite at room temperature, and that nickel, manganese, and chromium, are next in this respect, in the order given. (Nitrogen is probably also extremely effective.) The strong carbide forming elements molybdenum, tungsten, and vanadium are less effective in causing austenite to be retained at room temperature because they tend to prevent carbon from being dissolved in the austenite.

Summary and Comments

It has been shown that a quench-temper procedure is a relatively simple method of following the progress of austenite transformations at low temperatures. It was found that a variety of tool steels, from plain carbon, to highly alloyed high speed steels, harden in the same manner, that is, their austenites transform to martensite gradually as the temperature decreases from about 400 to 80 degrees Fahr. (205 to 25 degrees Cent.). The rate of cooling through this range of temperatures has no effect on the formation of martensite, since the progress of the reaction depends only on decrease in temperature, and not on time at these low temperatures. Isothermal reactions also may occur at temperatures below 400 degrees Fahr. (205 degrees Cent.) but they are rarely encountered in ordinary heat treatment. Since the formation of martensite depends on the continuous cooling of austenite down to temperatures below 200 degrees Fahr. (95 degrees Cent.), it is faulty heat treating practice to transfer tool steels to the tempering furnace before they have been cooled in the quench to about 100 to 150 degrees Fahr. (40 to 65 degrees Cent.). Very high austenitizing temperatures may cause the retention of austenite at room temperature in some of these steels. This effect of high temperatures is due mainly to the solution of carbon and alloying elements in the austenite and not to austenite grain size.

Many of the phenomena discussed above have been known to skilled tool hardeners for many years. If this paper helps to decrease the apprentice period for those less skilled, the authors will consider their efforts truly rewarded.

ACKNOWLEDGMENTS

The authors are grateful to the executives of the Crucible Steel Company of America for permission to present the data obtained at the Eastern Research Laboratory. To Mr. H. J. Stagg of Halcomb Steel Division, without whose enthusiastic encouragement and support this paper would not have been written, the authors extend their sincere thanks. Many of the men on the staff of the Research Laboratory have aided in the preparation of the paper, and the authors wish to express their apppreciation to all of these, and particularly to Messrs. F. Baureis, P. P. Hydrean, C. H. Savage, and G. Stefanelli.

Bibliography

1. E. S. Davenport, "Isothermal Transformation in Steels," TRANSACTIONS, American Society for Metals, Vol. 27, 1939, p. 837.
2. P. Payson, W. L. Hodapp, and J. Leeder, "The Spheroidizing of Steel by Isothermal Transformation," TRANSACTIONS, American Society for Metals, Vol. 28, 1940, p. 306.
3. J. L. Ham, R. M. Parke and A. J. Herzig, "Kinetics and Reaction Products of the Isothermal Transformation of a 6 Per Cent Tungsten-6 Per Cent Molybdenum High Speed Steel," TRANSACTIONS, American Society for Metals, Vol. 29, 1941, p. 623.
4. A. B. Greninger and A. R. Troiano, "Kinetics of the Austenite to Martensite Transformation in Steel," TRANSACTIONS, American Society for Metals, Vol. 28, 1940, p. 537.
5. E. R. Saunders and J. F. Kahl, "A Study of Martensite Formation by a Photometric Method," TRANSACTIONS, American Society for Metals, Vol. 30, 1942, p. 1139.
6. H. H. Chiswik, Discussion of "The Effect of Molybdenum on the Isothermal Subcritical Transformation of Austenite in Low and Medium Carbon Steels," TRANSACTIONS, American Society for Metals, Vol. 29, 1941, p. 336.
7. A. B. Greninger, "The Martensite Thermal Arrest in Iron-Carbon Alloys and Plain Carbon Steels," TRANSACTIONS, American Society for Metals, Vol. 30, 1942, p. 1.
8. Morris Cohen, Discussion of Reference 4.
9. B. H. DeLong and F. R. Palmer, "What Happens When High Speed Steel is Quenched," TRANSACTIONS, American Society for Steel Treating, Vol. 13, 1928, p. 420.
10. A.S.M. Metals Handbook, 1939 edition, p. 998, 1001, 1003, 1011, 1012.
11. A.S.M. Metals Handbook, 1939 edition, p. 989.
12. F. R. Palmer, "Tool Steel Simplified," 1937 edition, p. 285.
13. Morris Cohen and P. K. Koh, "The Tempering of High Speed Steel," TRANSACTIONS, American Society for Metals, Vol. 27, 1939, p. 1015.
14. A. Gulyaev, "Decomposition of Austenite in High Speed Steel at Constant Temperature," *Metallurg*, Vol. 15, No. 9, 1940, p. 43.
15. M. P. Gordon and Morris Cohen, "The Transformation of Retained Austenite in High Speed Steel at Subatmospheric Temperatures," TRANSACTIONS, American Society for Metals, Vol. 30, 1942, p. 569.
16. M. A. Grossmann and R. L. Stephenson, "The Effect of Grain Size on Hardenability," TRANSACTIONS, American Society for Metals, Vol. 29, 1941, p. 1.

17. E. S. Davenport, R. A. Grange, and R. J. Hafsten, "Influence of Austenite Grain Size upon Isothermal Behavior of S.A.E. 4140 Steel," *Transactions,* American Institute of Mining and Metallurgical Engineers, Vol. 145, 1941, p. 301.

DISCUSSION

Written Discussion: By Alden B. Greninger, General Electric Company, Lamp Department, Cleveland.

I congratulate Messrs. Payson and Klein on a remarkably clear write-up, showing the intimate relationship that exists between the kinetics of martensite formation and the practical heat treatment of tool steels. It is naturally gratifying to find in this paper, and in other papers presented at these conventions, results that are in complete agreement with the interpretation of martensite kinetics advanced by Dr. Troiano and myself at the 1939 meeting of this Society.[1] Also worthy of note is the fact that no evidence at all has appeared to support the "S-curve" interpretation of martensite formation proposed by the U. S. Steel Corp. Research Laboratory. Since that time, "S-curves" have continued to appear in the literature, but in these publications discussion of martensite formation has been avoided or passed over lightly, as though the subject were one of minor importance in both theory and practice of heat treating. It is encouraging, therefore, to see that some centers of metallurgical research have taken the opposite attitude. Certainly, it is difficult to see how our understanding of heat treatment can really progress without a good portion of our attention being devoted to the heart of the hardening process—martensite formation.

A matter of utmost importance in tool steel heat treatment concerns the temperature to which a given tool should be allowed to cool in the quench before it is tempered. It is true that many experienced heat treaters today follow the rule of never allowing the steel "to grow stone cold in the quench." This rule, which developed from experience and thus undoubtedly has some factual basis, at least for certain types of steel, should be amenable to rationalization, and eventually may be discarded, once it has been subjected to proper scientific study. The potential dangers involved in this practice should be obvious, and Messrs. Payson and Klein have logically stressed the point that ". . . . cracking is frequently due to the fact that the quenched steel is put into the tempering furnace, or bath, too soon, rather than not soon enough."

Attention should be called to a discrepancy between some of the authors' results and those of Gordon, Cohen, and Rose also reported at this meeting.[2] Both papers contain transformation curves for 18-4-1 high speed steel; Payson and Klein report only 2 per cent austenite for specimens quenched to room temperature, whereas Gordon and co-authors report approximately 25 per cent austenite for the same heat treatment. (On the other hand, both report about 40 per cent austenite for 200 degrees Fahr.)

Written Discussion: By B. F. Shepherd, chief metallurgist, Ingersoll-Rand Co., Phillipsburg, N. J.

[1] A. B. Greninger and A. R. Troiano, "Kinetics of the Austenite to Martensite Transformation in Steel," TRANSACTIONS, American Society for Metals, Vol. 28, 1940, p. 537.

[2] P. Gordon, M. Cohen and R. Rose, "The Kinetics of Austenite Decomposition in High Speed Steel," published on page 161 of this issue of TRANSACTIONS, American Society for Metals.

The formation of martensite and accompanying volume changes produce stresses which often materially lower the resistance to service or cause failure in the hardening operation. High austenitizing temperatures promote failure by cracking during quenching. The authors show in Tables III and IV that these high temperatures result in formation of the major percentage of the martensite at much lower than the initial temperatures where the greater rigidity probably assists in producing failures in quenching.

These hardening stresses are also greatly intensified by quick cooling through the range of 400 degrees Fahr. (205 degrees Cent.) to atmosphere. The authors show in Table I that a 0.035-inch thick specimen quenched from 550 degrees Fahr. (290 degrees Cent.) takes 10 seconds to cool to 80 degrees Fahr. (26.7 degrees Cent.) in oil and only 2½ seconds to cool to the same temperature in brine or water. A ¼-inch thick specimen quenched from 600 degrees Fahr. (315 degrees Cent.) takes 18 seconds to cool to 200 degrees Fahr. (95 degrees Cent.) in oil and 5 seconds to cool to 80 degrees Fahr. (26.7 degrees Cent.) in water. These represent considerable differences in cooling time through the range of formation of martensite and make it easy to understand the effect of cooling rapidly through this range on heavier sections.

These small sections were equalized throughout by holding at 550 and 600 degrees Fahr. (290 and 315 degrees Cent.). In normal practice, cooling direct from the austenitizing temperature results in greater temperature differentials. The old rule of selecting the slowest quench that will produce the desired hardness has a good solid foundation.

We have been normally limited to the use of brine and oil for the production of fully martensitic structures. These quenching mediums and methods limit the use of alloys to combinations which will cool faster than their critical cooling rate in their heavier sections and not form high residual hardening stresses.

We have been most liberal in the amounts and combinations of alloys used to slow up the critical cooling rate. The scarcity of these strategic materials has resulted in the development of the National Emergency Steels and the greater utilization of manganese and our most effective hardening element—carbon. These latter two elements have a great tendency with normal quenching methods to increase residual hardening stresses. I look forward to the still greater utilization of carbon with very low percentages of alloys and a quenching method which will consist of cooling the steel in molten salt at a temperature slightly above the temperature at which the initial formation of martensite occurs, equalizing for a length of time dependent upon the mass of the section followed by cooling in atmosphere. This will permit the martensite to form under conditions which will produce the lowest residual hardening stresses. The end results may be the use of very high carbon contents and very lean percentages of alloy.

It is difficult to believe that the extra tool steel and nondeforming steel have no austenite after normal quenching. It has been my belief that appreciable quantities of residual austenite remain in these steels. The presence of these small areas of very ductile material provides a cushion, capacity for adjustment and stress relief during the changes in volume, which occur during the

formation of the martensite grains, and permits the part to be hardened and removed from the quenching bath in one piece.

The increasing use of the term "austenitizing or austenizing (I prefer the latter because of its brevity) temperature" instead of "hardening temperature" is to be commended and it is hoped that the authors' practice in this respect will be followed.

Written Discussion: By W. R. Frazer, metallurgist, Union Twist Drill Co., Athol, Mass.

The authors of this excellent paper have clearly shown the similarity in hardening reactions between the simple straight carbon tool steels and the more complex highly alloyed high speed steels. The transformation curves for 18-4-1 and 5-4-1 high speed steel show that the austenite in 5-4-1 is much more stable than that in 18-4-1. This is also confirmed by the transformation curves developed by Gordon, Cohen and Rose in their current paper entitled "The Kinetics of Austenite Decomposition in High Speed Steel".

We were particularly interested in the study the authors made regarding the effect of austenitizing treatment on the formation of martensite. The data in Tables III to VII show that up to the temperature when maximum carbide solution takes place, there is a continuous change in the chemical composition of the austenite which has the effect of depressing the M-point or in other words, the austenite is stabilized as it is enriched with carbon and other solubles. After this point is passed, the grain growth that takes place does not have any effect on the formation of martensite because there is no chemical change. However, the coarse grain will certainly affect the toughness of the steel under impact.

This interesting study clearly brings out two fundamental don'ts for the hardener to follow:

1. Do not overheat.
2. Do not temper a tool you can't pick up with bare hands.

Written Discussion: By G. A. Roberts, metallurgist, Vanadium-Alloys Steel Co., Latrobe, Pa.

The presentation of transformation-temperature-time curves applicable to a number of common tool steel analyses is indeed welcome. The authors have presented information which represents a tremendous volume of experimental work and are to be congratulated on the emphasis placed upon the austenite to martensite transformation, particularly for these high carbon and high alloy steels.

The quantitative data concerning this transformation, however, cannot go unchallenged for it appears that the amounts of martensite existing at each temperature as indicated on the diagrams are somewhat in error. It is realized, of course, that the most important single factor influencing the M-point and the subsequent transformation to martensite on cooling in any one steel is the austenitizing temperature and the attendant change in the amount of carbon and alloying elements in solid solution. A decrease in the austenitizing temperature would materially increase the amount of martensite formed at any one temperature by raising the M-point if excess carbide still exists at the time of quenching. Even considering this, however, the results presented here, partic-

ularly for the amount of retained austenite at room temperature, are not consistent with a mass of information appearing in the literature.

The TTT curve for 18-4-1 high speed steel indicates that at room temperature 98 per cent martensite exists, that is, 2 per cent austenite is retained after quenching from 2350 degrees Fahr. (1290 degrees Cent.). Gordon, Cohen and Rose (page 161 this issue) offer data showing that 20 to 25 per cent austenite remains after such a treatment; Gulyaev and other Russian investigators have indicated in many publications that from 25 to 40 per cent austenite can be retained under similar conditions. The magnitude of many property changes obtained in this type of steel on tempering cannot be explained on the basis of as little as 2 per cent austenite.

Zmeskal and Cohen in 1942 Preprint 31 offer data showing 20 to 30 per cent retained austenite, depending on the cooling rate, in an air hardening high carbon-high chromium steel quenched from 1800 degrees Fahr. (980 degrees Cent.), whereas the authors have indicated that austenite is completely transformed to martensite in this steel at a temperature of around 150 degrees Fahr. (65 degrees Cent.) on cooling. Gulyaev again has shown large amounts of retained austenite in these steels treated by commercial procedures. Examination of the other curves presented also indicates a similar inherent error in the amounts of martensite formed.

It appears that this error is inherent in the technique used. The authors state that the tempering temperature was selected to eliminate any possibility of further transformation of the austenite, but this has not necessarily been achieved. It is to be noted that the time-temperature relationships used in the quench-temper treatment for high speed steel were 1 hour at 1050 degrees Fahr. (565 degrees Cent.) and for the high carbon-high chromium steel were 30 minutes at 1050 degrees Fahr. (565 degrees Cent.). These were selected presumably because the initial austenite did not transform isothermally at these temperatures on direct cooling until after some 48 to 60 hours but the retained austenite evidently acts differently and after holding a quenched high speed steel for 1 hour at 1050 degrees Fahr. (565 degrees Cent.), the austenite is so conditioned by precipitation of alloy carbides that it transforms to martensite on cooling, starting between 300 and 400 degrees Fahr. (150 and 205 degrees Cent.). Similarly, Zmeskal and Cohen show that the retained austenite in a high carbon-high chromium steel, oil-quenched from 1800 degrees Fahr. (980 degrees Cent.), is approximately 80 per cent transformed on cooling after holding for 30 minutes at 1050 degrees Fahr. (565 degrees Cent.). Because carbide precipitation precedes this, and considering the rapidity of etching of these samples, it seems reasonable that this newly formed martensite might escape detection from the original martensite.

It is also possible that an error is introduced in the metallographic technique used. Although the structural disposition of the retained austenite in steels is not known, it is probable that much of it must be located between the martensite needles, probably in a very finely divided form when high percentages of martensite exist. If this is true, tempering the martensite so that it will darken on etching would very probably obliterate all traces of the retained austenite. Although the authors do not claim any great accuracy for their quantitative measurements of the amounts of retained austenite, it would

seem that up to approximately 70 per cent transformation the metallographic technique might be satisfactory but when the major portion of the structure is martensitic, sensitivity is lost.

Written Discussion: By Morris Cohen, associate professor of physical metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

The writer is gratified to note that the modified type of S-curve diagram which he suggested three years ago[3] has been adopted in the present paper. The authors deserve considerable credit for demonstrating that this newer diagram, which portrays the fundamental aspects of the austenite-martensite transformation more adequately than the older type, is also helpful in explaining the practical heat treatment of tool steels. However, recent work at M. I. T. has shown that in high speed steels[4] and in high carbon-high chromium steels[5] the horizontal lines in the martensitic range are actually not quite horizontal. They slope upward to the right slightly, due to the isothermal formation of a small amount of bainite. This bainite seems to be nucleated by the presence of the martensite formed during the quench to the holding temperature, and appears long before the general formation of bainite at the time indicated by the C-curves.

In discussing the present paper, Rose and Gordon have pointed out that the metallographic technique employed by Payson and Klein may yield erroneous impressions of the amount of retained austenite in high speed steel. This also holds for the 1 per cent carbon-5 per cent chromium and the 1.5 per cent carbon-12 per cent chromium steels. When martensite, darkened by tempering, is the predominant phase in these alloys, the white and dark constituents are intermixed on such a fine scale that they are not clearly differentiated by the etching reaction. In fact, the dark-etching effects tend to spread across to the constituents which, under other conditions, would be expected to remain unattacked. Such obscuration may be further accentuated by the photographic process used to reproduce the structure. Both of these factors help explain why the authors' estimates of retained austenite undoubtedly lie in the low-side of the true values.

In a recent paper, Zmeskal and Cohen[6] studied the transformation characteristics of retained austenite during the tempering of 1 per cent carbon-5 per cent chromium and 1.5 per cent carbon-12 per cent chromium steels after austenitizing at 1700 to 2200 degrees Fahr. (925 to 1205 degrees Cent.). It is therefore somewhat disconcerting to note that Payson and Klein find no retained austenite in the latter steel and only 5 per cent in the former steel after austenitizing at 1800 and 1850 degrees Fahr. (980 and 1010 degrees Cent.) respectively. Accordingly, the writer had specimens of the two steels water-quenched and air-cooled from 1800 and 1850 degrees Fahr. (980 and 1010 degrees Cent.). The chemical analyses were as follows:

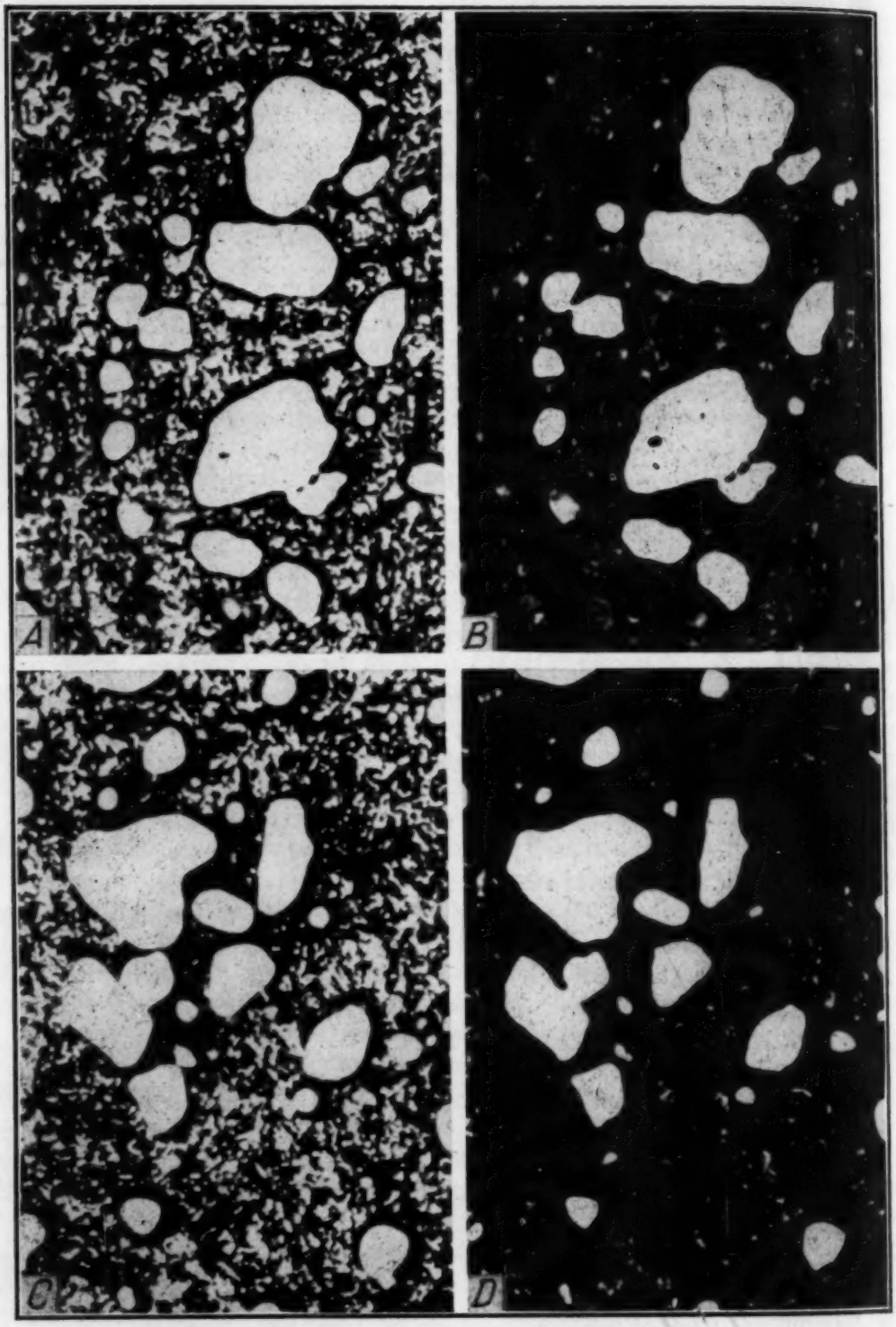
	Carbon	Chromium	Vanadium	Molybdenum
1 Per Cent Carbon- 5 Per Cent Chromium	1.00	5.31	0.27	1.13
1.5 Per Cent Carbon-12 Per Cent Chromium	1.60	11.95	0.25	0.79

[3]M. Cohen, Discussion of paper by A. B. Greninger and A. R. Troiano, TRANSACTIONS, American Society for Metals, Vol. 28, 1940, p. 537.

[4]P. Gordon, M. Cohen, and R. S. Rose, page 161 this issue.

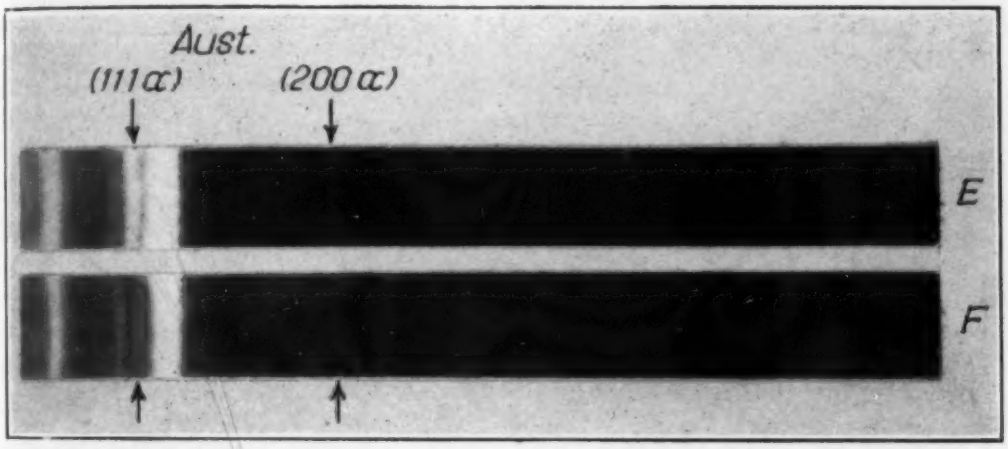
[5]D. P. Antia, Massachusetts Institute of Technology, Cambridge, Mass. Unpublished research.

[6]O. Zmeskal and M. Cohen, American Society for Metals, Preprint 31, 1942.



Figs. A-D—Microstructure of 1.5 Per Cent Carbon-12 Per Cent Chromium Steel, Austenitized at 1800 Degrees Fahr., Cooled to Room Temperature, Tempered at 1050 Degrees Fahr. for 30 Minutes, and Water Quenched to Room Temperature. Etched With 12 Per Cent Nital. × 2000. Fig. A—Air Hardened. Focused on Matrix. Fig. B—Air Hardened. Focused on Carbides. Fig. C—Water Hardened. Focused on Matrix. Fig. D—Water Hardened. Focused on Carbides.

and thus the steels were quite similar to those studied by Payson and Klein. After darkening the martensite in these hardened steels by tempering as recommended by the authors, the structures were examined microscopically. Prints of the 1.5 per cent carbon-12 per cent chromium steel are shown in Figs. A to D. The pictures in Figs. A and C were taken by focusing on the matrix, and a considerable amount of white appears in the background. This white is due to the presence of retained austenite, but is too finely dispersed to permit even an approximate estimate of its absolute amount. Despite the similarity of Figs. A and C, more austenite is retained on air cooling than on water quenching. This effect was clearly demonstrated by Zmeskal and Cohen[6] as well as by the diffraction patterns of Figs. E and F. Hence in steels of this type, even if the authors were able to obtain an accurate determination of the retained austenite content after hot quenching to low temperatures approaching room temperature, the results would still not apply to commercial practice where air hardening is used.



Figs. E and F—X-Ray Photograms of 1.5 Per Cent Carbon-12 Per Cent Chromium Steel After (E) Air Hardening and (F) Water Hardening from 1800 Degrees Fahr. Intermediate Phragmen Camera, Chromium Radiation. Note Definite Austenite Lines.

Figs. B and D were taken on the same spots as Figs. A and C respectively, but the focusing was done on the carbides. Obviously, the matrix now prints much blacker, and appears to contain much less austenite. The X-ray photograms in Figs. E and F show indisputably the existence of appreciable quantities of retained austenite in the 12 per cent chromium steel, both after water and air hardening from 1800 degrees Fahr. (980 degrees Cent.).

The presence of much more retained austenite can be shown by the same methods after hardening the 5 per cent chromium steel from 1850 degrees Fahr. (1010 degrees Cent.). Again, air hardening causes the retention of more austenite than water hardening.

In the light of these findings, it is hoped that the authors will review in detail the many steps entering into their metallographic estimates of retained austenite in the high chromium and high speed steels. In alloys of this type, such variables as depth of etching, method of focusing, resolving power, magnification, exposure and developing conditions, all have their influence on the

appearance of the microstructure in the final prints. It should be emphasized, however, that in many of the simpler steels, a clear-cut delineation of the tempered martensite and retained austenite may be obtained, and in such cases, the determination of the retained austenite by the metallographic methods is applicable, even when the amount of retained austenite is less than 5 per cent.

Written Discussion: By Paul Gordon, research assistant, Massachusetts Institute of Technology, Cambridge, Mass., and Robert S. Rose, sales metallurgist, Vanadium-Alloys Steel Co., Boston.

This paper by Payson and Klein is of particular interest to us, since during the past year we have been studying the transformation rates and decomposition products of high speed steel. Comment will, therefore, be mainly directed to that section of this paper dealing with these types.

The implication of Fig. 9 is that the matrix of 18-4-1 quenched from 2350 degrees Fahr. (1290 degrees Cent.) to room temperature consists of 98 per cent martensite and 2 per cent residual austenite. We believe the quantity of austenite retained under these conditions to be greater, indeed, several times greater, and most likely in the vicinity of 20 per cent. In addition to the direct approach of calibrating the expansion which would accompany 100 per cent transformation, and deriving percentage from the actual observed dilation, there are several other manifestations of residual austenite in high speed steel that can be measured quite accurately. The increase in specific volume attendant to the tempering of quenched 18-4-1 can be associated only with the transformation of residual austenite. The measured increase between a tempering temperature of 600 and 1050 degrees Fahr. (315 and 565 degrees Cent.) is 0.00045 c.c. per gram. If this enlargement is the result of the decomposition of only 2 per cent austenite (the amount indicated by Payson and Klein), then complete transformation, i.e., the change from the primary austenitic to the quenched and fully tempered state, would effect an expansion of 0.0225 c.c. per gram equivalent to approximately 21 per cent. S. G. Fletcher[7] and A. B. Greninger[7], however, have independently estimated the change in volume accompanying the gamma-alpha transformation in straight carbon steel as 3.5 to 4 per cent, which is undoubtedly high for 18-4-1 due to the volume occupied by excess carbide. Even with this figure, however, it is evident that the increase in volume on the basis of 2 per cent retained austenite is 5 to 6 times the amount observed by Fletcher and by Greninger. We doubt that many high speed tools could accommodate themselves to a 21 per cent increase in volume during the hardening operation. Likewise, the dilation that occurs during cooling from the tempering heat is explained only by the decomposition of residual austenite. If this dilation of 0.0015 inch per inch is the result of conversion of only 2 per cent austenite, 100 per cent transformation would amount to 0.075 inch per inch equivalent to 7.5 linear and 22.5 per cent volume expansion. This is also approximately six times the observed increase in volume for complete transformation. Moreover, the indications of both X-ray and magnetic analysis are that the quantity of residual austenite in quenched 18-4-1 is much greater than 2 per cent.

Despite the authors' statement on page 226 that the photomicrographs of

[7]Personal Communication.

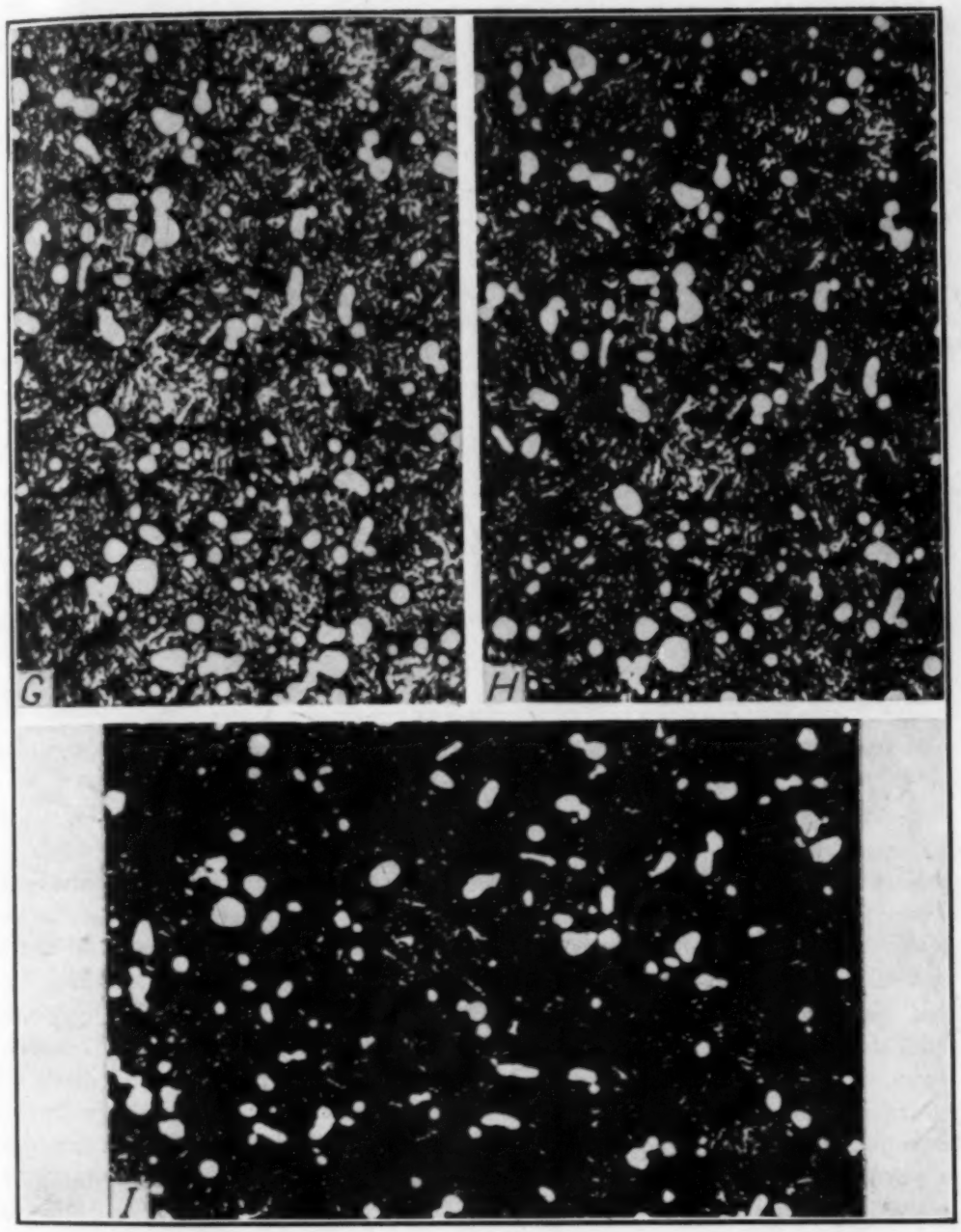


Fig. G—Photomicrograph of High Speed Steel 18-4-1. × 1000. Nital Etch. Printing Exposure, 3 Seconds.

Fig. H—Photomicrograph of High Speed Steel 18-4-1. × 1000. Nital Etch. Printing Exposure, 5 Seconds.

Fig. I—Photomicrograph of High Speed Steel 18-4-1. × 1000. Nital Etch. Printing Exposure, 15 Seconds.

18-4-1 on page 238 show their method of estimating percentages of transformed product, we fail to find any explanation of the technique applied to the photomicrographs that permitted even semiquantitative estimations. Actually, in high speed steel, when the ratio of phases is high, and particularly when the phase

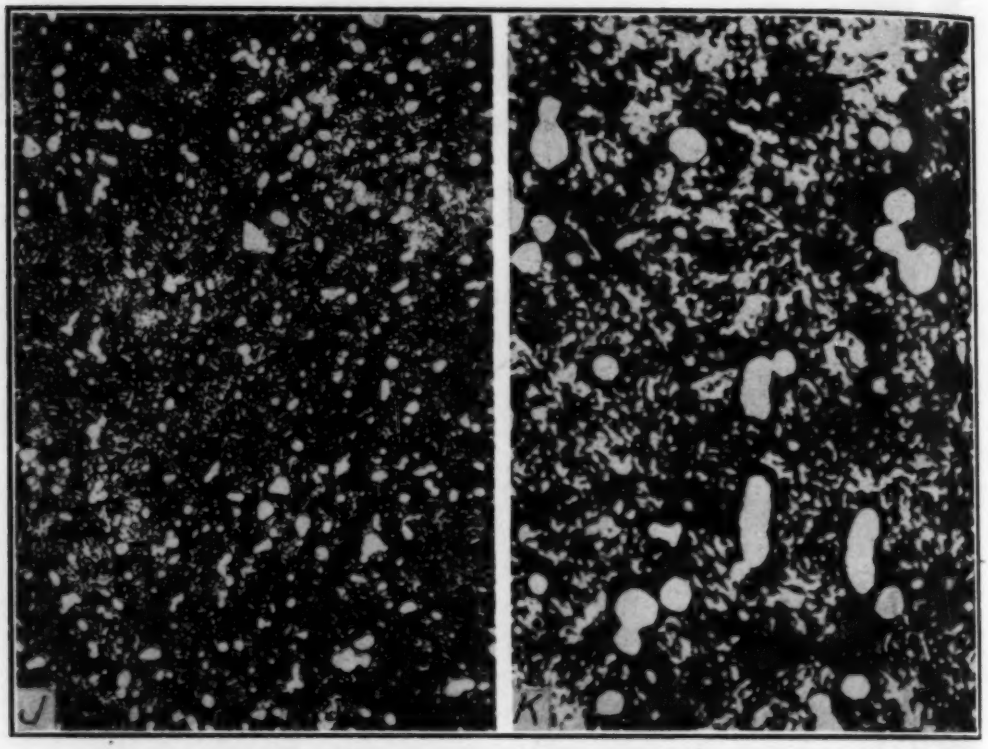


Fig. J—Photomicrograph of High Speed Steel 18-4-1. × 500. Nital Etch. Printing Exposure, 5 Seconds.

Fig. K—Photomicrograph of High Speed Steel 18-4-1. × 2000. Nital Etch. Printing Exposure, 5 Seconds.

present in the smaller amount is finely dispersed, metallographic analysis, it seems to us, is incapable of yielding accurate percentage data. An examination of Fig. 21 reveals many blurred and streaky spots that we feel are conditioned by the etch, the magnification, and the printing methods. Figs. G, H and I of this discussion are not only from the same specimen, but are actually prints from the same plate. They were exposed in printing 3, 5 and 15 seconds respectively. The obvious difference in quantity of the finely dispersed white particles emphasizes the uncertainty and variation most likely to accrue from the metallographic method. This error does not arise when the untransformed austenite is relatively large in quantity and massive in form. The effect of variation in magnification is illustrated by Figs. H, J and K, which are at 500, 1000 and 2000, respectively.

It is to the fallibility of the metallographic method when applied to high speed steel quenched to below 200 degrees Fahr. (95 degrees Cent.) that we attribute the authors' very low estimate of retained austenite at these temperatures.

Authors' Reply

It is gratifying to us to find that this paper has brought forth so much animated discussion.

We heartily agree with Dr. Greninger that all transformation-temperature-

time curves should show the progress of the austenite-martensite transformation so that the reactions which occur during the hardening of the steel can be more thoroughly understood. We hope that all curves published in the future will include this important practical feature, and in spite of the discussion to the contrary, we believe that the method of Greninger and Troiano can readily and satisfactorily be used to obtain these data.

We are pleased that Mr. Shepherd and Dr. Frazer, both of whom have had many years of experience in the heat treatment of tool steels, find our data useful in interpreting the behaviors of these steels when they are hardened. Mr. Shepherd offers the extremely interesting suggestion that the resistance of steels to cracking in the quench is due to the presence of some ductile material which acts as a cushion to absorb the volume changes which occur during the formation of martensite. We believe that in relatively shallow hardening steels this cushion consists of high temperature transformation product, the so-called unhardened core, rather than retained austenite. We show in Tables III and IV that Extra and Oil Hardening steels have more austenite retained at room temperature, when they are austenitized at 1600 degrees Fahr. (870 degrees Cent.), than when they are austenitized at 1450 degrees Fahr. (790 degrees Cent.), and yet these steels are much less likely to crack when they are austenitized at 1450 degrees Fahr. (790 degrees Cent.) than when a higher austenitizing temperature is used.

Dr. Roberts, Dr. Cohen, and Messrs. Gordon and Rose chide us for stating that the higher alloy steels contain so little austenite retained at room temperature. We had tried to forestall such discussion by our statements in the upper half of page 226 of the paper, to the effect that the percentages indicated on these curves should not be taken too literally. We repeat here that we made no attempt to measure precisely the amounts of austenite retained at low temperatures. We should have made it more clear that all our estimates were strictly estimates, and not measurements. The statement that 98 per cent austenite had transformed at some temperature means that a small amount of light etching structure was visible in the microstructure and that this amount was about 2 per cent. The amount might have been 5 per cent—it could not have been as much as 20 per cent.

Dr. Roberts contends that the determination of retained austenite by the Greninger-Troiano procedure is not reliable because "error is inherent in the technique used". We definitely disagree with this opinion. We tried to make it clear in Fig. 1 that the martensite which forms initially from the austenite is distinguished from the martensite which forms eventually from the residual austenite, in that the former, having been tempered, etches dark, while the latter, which has not been tempered, etches light. Even if the residual austenite in the high speed steel was conditioned by the heating at 1050 degrees Fahr. (565 degrees Cent.) for 1 hour, to transform during the subsequent cooling, as Dr. Roberts surmises, the martensite which resulted could readily be distinguished from the tempered product of the original transformation because of the characteristic difference in the etching effect on untempered, and on tempered, martensite.

Dr. Cohen and Messrs. Gordon and Rose emphasize by their photomicro-

graphs that improper printing technique may lead to improper conclusions. We do not quarrel with this opinion, but it is not relevant to our paper because we did not rely on photomicrographs for our estimates of the amounts of transformation products. Our estimates were based entirely on visual examination of many fields of microsections properly etched at × 1200 magnification. The photomicrographs in the paper were submitted merely as illustrations of changes in relative amounts of tempered and untempered martensite as the austenite-to-martensite reaction proceeds.

We have great respect for Dr. Cohen's work, and we are willing to concede that our estimates of the quantitative progress of transformation of the high alloy steels at low temperatures may be in error since they do not agree with the much more precise measurements made by Dr. Cohen on similar steels. However, if we have erred, we have erred on the conservative side from the viewpoint of the tool hardener. We have emphasized that it is necessary for tool steel to be cooled to approximately 125 to 150 degrees Fahr. (52 to 66 degrees Cent.) during the quench in order to permit most of the austenite to transform. If, as has been contended, our estimates of the amounts of austenite present in tool steels at, say, 175 degrees Fahr. (78 degrees Cent.) are on the low side, then there is even greater reason for our recommendation. We are interested primarily in explaining why tool steels must be cooled to low temperatures before they are tempered. Whether or not the amount of austenite retained at room temperature in high speed steel is 2 or 10 per cent we believe to be of secondary importance.